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British Association for the Advancement of Science

FIFTH REPORT

COLLOID CHEMISTRY

AND ITS

GENERAL AND INDUSTRIAL APPLICATIONS

(With Index to the Five Reports)

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PREFATORY NOTE

At the request of the British Association for the Advancement of Science, the Department of Scientific and Industrial Research has decided to extend to the Fifth Report on Colloid Chemistry and its General and Industrial Application, the last of the series, the arrangements under which the earlier reports of the series were published for the Department of H M Stationery Office The preparation of the Fifth Report like that of its predecessors has been entirely the work of the Committee of the Association, and the Department of Scientific and Industrial Research is in no way responsible for the statements of opinion or of fact contained in it The share of the Department in the work has been limited to arranging for the publication of the Report, in order to ensure that the information given therein may quickly be made available

To make the information contained in these five Reports more easily accessible, a general index to the whole series has been prepared by the Department, with the concurrence of the Association, and is included in the present volume

Department of Scientific and Industrial Research
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November, 1923

Colloid Chemistry and its General and Industrial Applications — *Fifth Report of the Committee, consisting of* Professor F. G DONNAN (*Chairman*), Dr W CLAYTON (*Secretary*), Dr E ARDERN, Dr E F ARMSTRONG, Sir W M BAYLISS, Professor C H DESCH, Dr A E DUNSTAN, Mr H W GREENWOOD, Mr W HARRISON, Mr E HATSCHEK, Mr G KING, Professors W C McLEWIS and J W McBAIN, Dr R S MORELL, Professors H R PROCTER and W RAMSDEN, Sir E J RUSSELL, Mr A B SEARLE, Dr S A SHORTER, Dr R E SLADE, Mr F SPROXTON, Dr H P STEVENS, Mr H B STOCKS, Mr R WHYMPER

INTRODUCTION

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The Committee again expresses its deep sense of obligation to the gentlemen who have compiled the several papers constituting this Report Special thanks are due to the Department of Scientific and Industrial Research for the preparation of an Index to the Committee's five Reports

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June, 1923

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THE MEASUREMENT OF SURFACE TENSIONS

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(*East London College*)

Received February, 1922

It may save repetition if I here define the various symbols used throughout this report

T = surface tension, θ = temperature, m = mass, g = the acceleration due to gravity, h = the height of ascent of a liquid in a capillary tube, r = radius—usually the radius of a capillary tube, or the radius of maximum section of a large drop or bubble, R = a radius of curvature—usually the radius of curvature at the vertex of a capillary surface, γ = contact angle, a^2 ($\equiv T/gp$) stands for the so-called specific cohesion of a liquid Numerical values of T are always given in dyne-cm^{-1}

This report deals with recent advances in the methods and technique used in the determination of surface tensions, and it advisedly concerns itself with methods rather than results It is now clearly recognised that an accurate knowledge of the tension in surfaces separating a liquid from a vapour phase, and, more especially, of the tension in a liquid-liquid or a liquid-solid interface, is a first condition for the quantitative discussion of many of the problems of colloid chemistry and physics The methods for the determination of these latter constants are barely emerging from a primitive stage of development, and it cannot be said that a standard method for their measurement has yet been evolved It is important, therefore, to discuss the methods of measuring surface tensions at a liquid-vapour interface, for, apart from the interest which the results of these methods may have to the worker in colloids, a discussion of the more important of the later advances in these methods will furnish ideas which may serve in the attack on the more difficult problem of the measurement of interfacial tensions

Border-line subject as it is, the measurement of surface tension has attracted the attention of chemists, physicists and biologists, and, while the intellectual interest imported into the subject by workers who look at it from very different angles is all to the good, there are none the less important disadvantages to be set down on the debit side Of these, a lack of appreciation of the mechanical principles involved in the establishment of the equations of equilibrium on which the measurements depend, is undoubtedly one of the most serious, and it is still not uncommon to find in modern papers and treatises statements of the type "Surface tension is defined as the force which acts *at right angles to the surface of a liquid** along a line of unit length", "the ideal weight of a falling drop is given by $2\pi rT$", "the drop falls when its weight just exceeds the surface tension"; "the acceleration due to gravity is 981 *dynes* at"—Weiss-nichtwo, shall we say? And the like Surface tensions are expressed

* Italics mine, this astonishing definition is to be found in a recent edition of a text-book published for the use of pass and honours students in the Universities

in all sorts of units, correct and incorrect—dynes, unfortunately, are sometimes given—and that unhappy quantity, molecular surface energy, has been credited with a goodly proportion of the units of the physical calendar "Large" and "small," as terms applied to the capillary surface, are not exactly appreciated, and, most important of all, in the use of those methods which depend on a knowledge of the contact angle, this angle is sometimes assumed to be zero on very slight evidence, sometimes is neglected on no evidence at all, so that the results given are of very doubtful value

The remarkably rapid development of colloid chemistry and physics during the last generation is reflected in a correspondingly large increase in the number of measurements of capillary constants, and it is gratifying to record that, especially in the last five or six years, a larger measure of attention has been given to some of the points outlined above But even now misstatements are still far from uncommon, and a knowledge of the magnitude of the contact angle being of the first importance, it will perhaps best serve the purposes of those who wish to make use of this report if I discuss, first, the methods lately employed for the determination of contact-angles, then the advances in the measurement of the surface tension at a liquid-vapour surface, and, finally, such methods as have been employed for the measurement of interfacial tensions

Contact angles

Probably the best method for obtaining the value of a contact angle is to measure the surface tension of a liquid by two different methods—one giving the value of T independently of the contact angle, while the second method depends on an equation involving both T and γ In this way each of the important capillary constants may be determined independently Magie[1], by measuring the total depth of a large bubble of air imprisoned beneath a concave lens immersed in the liquid, and by comparing this with what we may call the "semi-depth" of the bubble—the vertical distance between the vertex and the plane of greatest horizontal section—has determined a number of contact angles, and Langmuir[2] has also used this method I have elsewhere[3] classified a number of the more important methods for the measurement of surface tensions, the main grouping being into those (A) dependent on, and (B) independent of a knowledge of the contact angle Any pair of these methods may be chosen—we may, for example, combine the capillary-rise method, the weighing of the pull on a vertical plate, or the measurement of the pull on a spherical surface touching the liquid, from (A), with the weighing of the tension in a film, or the observation of the maximum pressure required to release a bubble from the orifice of a capillary plunged vertically into the liquid, from (B)

During recent years, a number of experiments have been made to determine contact angles directly, and, apart from an actual estimate of the magnitude of the contact angle, to determine whether it departs so appreciably from a zero value as seriously to affect the value of the surface tension determined on the assumption that the contact

angle is zero Pockels[4] has discussed the problem generally and—a matter of very great importance in these measurements—gives full instructions for ensuring the perfect cleanliness of the surface under observation

From the more prominent experiments may be cited those of Furth[5], who measures the angle of contact of mercury with glass from observations made on a convex surface partially covered with mercury Anderson and Bowen measure[6], by an optical method, the radius of curvature (R) at the vertex of the capillary surface inside a vertical glass tube Several observations are made, using tubes of different radii Remembering that the limiting value of r/R, as r approaches zero, is the cosine of the angle of contact, it is clear that if we plot values of r as abscissæ against those of R as ordinates the inclination of the tangent at the origin is given by $\tan \phi = \cos \gamma$ For water, glycerine, olive oil and turpentine the value of ϕ is very closely 45°, pointing to a zero contact angle with glass for each of these substances For mercury the contact angle with glass is found to be 139° These results have been confirmed for water, glycerine, olive oil and turpentine by the optical examination[7] of a long cylindrical drop of liquid pendent from a thin plate of glass held with its plane vertical Clark[8], measuring interfacial tensions by the capillary-rise method, determines γ approximately by comparing the depth of the meniscus with the radius of the tube The latest direct experiments on the subject are those due to Richards and Carver[9], who observe the angles of reflection of rays of light in the region immediately surrounding the line of union of the liquid with a vertical glass plate They find that the angle of contact between water and glass is very accurately equal to zero

The Surface Tension at a Liquid-gas Interface

The main advances made during the last ten years in these measurements are concerned with improvements in the technique of the capillary-rise method and of the drop-weight method

The chief troubles which beset observers who use the capillary-rise method are, apart from the necessity common to all surface-tension methods of working with a perfectly clean surface

(i) temperature control,
(ii) tube calibration,
(iii) small corrections,
(iv) contact angle difficulties

The difficulties connected with the measurement of the contact angle, so often slurred over, are, as we have seen in the last section, at last receiving something approaching to a due measure of recognition But, this apart, the technique of the capillary-rise method will always present serious difficulties, and unless time is no object, and apparatus can be elaborated at will, the method can hardly be considered as ideal The measurement and control of the temperature of the meniscus demand precise thermostatic and optical arrangements, and the selection, calibration and cleaning of a suitable tube have been known to take up some months of the time of an investigator

The measurement of the surface tension of highly viscous liquids presents problems which cannot yet be said to be satisfactorily solved Nisi[40] has determined the surface tension of glycerine, employing interference fringes to measure the curvature of the capillary surface, and Berggren,[41] by measurements made on a thread of copal varnish hanging vertically, has attempted to determine the surface tension of this substance

Interfacial Tensions

As previously mentioned, the measurement of interfacial tensions has barely emerged from its primitive state The last few years have, none the less, seen notable advances, and it is not too much to hope that the work of the next few years will produce figures for interfacial tensions which will prove to be as consistent as the recent numbers obtained for liquid-air tensions

The drop-weight (*not* the drop-number) method gives, in the hands of Harkins, very self-consistent results These will be discussed later, but we may here say that the pressing need of the moment is for a systematic series of determinations of some one interfacial tension by a number of independent and accurate statical methods A comparison of the results so obtained would soon point to the method or methods which would give dependable figures

In measurements of interfacial tensions we owe much to the pioneer work of Quincke[42] Many of his methods have been later subjected to modification, but the initial impetus in this, as in much other surface-tension work, is largely due to him

Many of the methods discussed in the preceding section have been used for interfacial work, and, unfortunately, much of the work has proved to be unreliable

Watson[43] has used the ripple method for the measurement of the tension in mercury-water, mercury-alcohol, benzene-water and petroleum water interfaces, using the equation

$$T = \frac{n^2\lambda^3}{2\pi}(\rho_1 + \rho_2) - \frac{g\lambda^2}{4\pi^2}(\rho_1 - \rho_2),$$

where n is the frequency of the dipping fork, and λ the wave-length of the ripples generated

Apart from the disadvantage that the equation is a cubic in λ the method, as applied to interfacial tensions, is distinctly difficult in practice Troubles due to upper-surface waves, and to reflection of light from this surface, may be remedied, but the marked damping of the interfacial waves makes the measurements both difficult and unreliable

For mercury-water Watson found values of T_0 varying between 385·8 and 364 1 For mercury-alcohol T_0 varied between 378 4 and 364 3 For water-benzole and for water-petroleum the mean values of some rather variable figures gave T_0 equal to 33 12 and 35 38 respectively Watson attempted to verify these latter figures by the use of a spring balance, measuring directly the tension in the thin film formed when a wire rectangle with its plane vertical is drawn up

from the interface For water-benzole he found a value 35 86, but the figure obtained by this method for water-petroleum was about 44, not at all in agreement with that obtained by the ripple method The discrepancy was ascribed to the difficulty of forming a true film, and an attempt to measure the interfacial tension in terms of the *maximum* pull experienced when a short horizontal rod is withdrawn from the interface met with greater, though still qualified, success

V Lerch[44] has used the capillary-rise method to measure the interfacial tension between water and benzene The capillary tube was immersed in a *wide* containing vessel, and the value obtained for T was 32 3

Clark[8] has also used the capillary-rise method to measure interfacial tensions, the capillary tube (internal radius $r = 0\ 060$ cm, external radius $r_2 = 0\ 088$ cm) being immersed vertically in a narrow tube of radius $r_3 = 0\ 279$ cm The tension was determined by the equation

$$T = \frac{g(\rho - \rho_1)h}{2\cos\gamma\left(\frac{1}{r_1} - \frac{1}{r_3 - r_2}\right)},$$

for which no proof is given His method for measuring γ has been previously described For alcohol-olive-oil he finds $T_{30} = 14\ 2$, for ethyl ether-glycerine $T_{10} = 15\ 4$

Lynde[45] has investigated the effect of pressure on interfacial tensions The denser of the two liquids was placed in a U-tube, having a narrow limb of 1 mm and a "wide" limb of 1 cm bore The tube was immersed in the lighter liquid and pressures varying between 1,000 and 6,000 pounds per square inch were applied to the whole system Calculations of the actual values of the surface tension were not made, the quantity $100/T\ \delta T/\delta p$ being calculated from the change in the capillary elevation For mercury-water and for mercury-ether systems this coefficient was constant and positive For ether-water and for chloroform-water systems the coefficient was constant and negative With carbon disulphide-water the coefficient was positive and varied slightly with the pressure No correction was applied for the radius of the wider tube, and the contact angle was assumed to be zero

By far the most accurate of the capillary-rise experiments are those due to Harkins and Humphery,[46] who used, as a "wide" vessel, a special container which did not necessitate the use of excessive quantities of liquid The technique of the method is admirable Water is poured into the container, and a vertical capillary tube is completely immersed therein Benzene is then poured on to the water surface, and the tube is raised by a ratchet until the water-benzene meniscus *falls* to some definite point The vertical distance between this point and the tip of a platinum pointer touching the interface in the container is carefully measured and the surface tension deduced in the usual way Their results (assuming the contact angle to be zero) are probably the most reliable yet obtained and are here given in extenso

BENZENE-WATER INTERFACE

T	θ (° C)
34 98	10°
34 52	20°
34 18	25°
33 82	30°
33 22	40°

These results, they say, are represented between 0° and 40° by

$$T = 35\ 54\ (1-0\ 056\theta),$$

which is presumably a misprint for

$$T = 35\ 54-0\ 056\theta$$

The capillary-rise method has lately been used in much more primitive fashion by Reynolds,[47 48] who places the capillary in a burette and measures the capillary rise in terms of the burette scale divisions The reduction of these readings to centimetres, the meniscus correction, and the correction due to the curvature of the interface outside the capillary tube are simultaneously made by a method whose validity is, apparently, nowhere investigated The result for a benzene-water interface ($T_{19} = 34\ 4$) is in fair agreement with that obtained by Harkins and Humphery, but not at all in agreement with some check experiments made by Reynolds using the dropping pipette These gave a value of 28 0 for the interfacial tension at 19° The discrepancy is hardly surprising, as the surface tensions are evaluated by means of a formula which is analogous to the equation

$$mg = 2\pi r T$$

for liquid-air tensions, and this formula, as we have seen, is completely invalid

Gouy[40] has determined the tension at a mercury-water interface by measuring with a travelling-microscope the dimensions of a large drop of mercury immersed in water For a freshly formed surface he obtains the value $T_{18} = 426\ 7$, and has noted that if the system be allowed to stand the surface tension remains for a time constant in value, and then begins to fall Lenkewitz[50] has used the same method, calculating the surface tension from measurements made directly on the ten-times-magnified image of a mercury drop about 7 cm in diameter

By far the most interesting results for interfacial tensions are those due to Harkins and his collaborators The importance of their work demands detailed treatment We have already mentioned that Harkins and Brown[29] have studied the variations in the value of the function F in

$$mg = 2\pi r T\ F\left(\frac{r}{a}\right)$$

Remembering that a has the dimensions of a length, we see that the equation

$$mg = 2\pi r T\ \varphi\left(\frac{r}{V_t}\right)$$

is dimensionally sound, where V is the volume of a detached drop Harkins and Brown, knowing from capillary-rise experiments the surface tensions against air of water and of benzene, carried out a most careful series of experiments similar to those initiated by Rayleigh, and discussed in the last section They plotted values of $F(r/a)$ against the corresponding values of r/a, and did the same for $r/V^{\frac{1}{3}}$ and the corresponding values of $\varphi(r/V^{\frac{1}{3}})$

One method used to evaluate a surface tension from the weight of a falling drop is to calculate V from the known mass of the drop and the density of the liquid, then to find $r/V^{\frac{1}{3}}$, and hence from the curve, the corresponding value of $\varphi(r/V^{\frac{1}{3}})$

The surface tension is then given by

$$T = \frac{Mg}{2\pi r\ \varphi\left(\frac{a}{V^{\frac{1}{3}}}\right)}$$

In measuring interfacial tensions, the volume of the detached drop is usually measured directly, being observed from the change in volume of the liquid in the pipette Harkins and Brown make the important observation that the natural period of fall of the drop should be five minutes or more, and point out that " if dilute aqueous solutions of long chain organic molecules are used it is often necessary to let the drop hang at full size for half an hour or more in order to obtain the static surface tension of the solution "

Such an observation as this makes it clear that the ordinary practice of the drop-number method is beset with pitfalls In the hands of Hardy[51] and of others it has yielded useful results, but it is not too much to say that many of the figures for interfacial tensions obtained by its aid are not only valueless but positively misleading It is, for example, quite useless to measure the slowness of formation of the drops by giving the number of cubic centimetres which escape from the pipette in a given time Until the volume of each drop is known, nothing can be deduced from such data Moreover, to obtain satisfactory results it is necessary to adjust the dimensions of the tip to suit the particular system under observation Harkins and Brown, for instance, record the formation of a drop having a volume of nearly *fourteen cubic centimetres*, when anisol and water were the liquids employed Nevertheless, a tip having a diameter of 9 5 mm gave quite satisfactory results

Harkins and Grafton[52] have used the drop-weight method to determine the tension at mercury-liquid interfaces For mercury-water they find $T_{20} = 374\ 8$,—a mean of several closely concordant numbers

The great variation in the values given by the various methods quoted for the magnitude to the tension at a mercury-water interface serves further to emphasise the need for more independent measurements It is not necessary here to elaborate favourable lines along which it seems desirable that future research should proceed—I have already discussed the matter elsewhere[16] in fairly

full detail It is sufficient here to say that several promising methods can be devised having as their basis the equation

$$p = \Gamma\left(\frac{1}{R_1} + \frac{1}{R_2}\right)$$

The pressure excess p at any point of the surface may be determined experimentally, and the principal radii of curvature (R_1 and R_2) at this point may be evaluated in several different ways,[16 53] a comparative study of which would well repay the trouble involved Photographs of large drops, or, indeed, of drops of any size, may be made, and measurements made thereon should yield the necessary information Jaeger's method may be suitably modified to meet th and the measurement of the *maximum* pull on an anchor slowly from the interface commends itself as a suital I am at present engaged in developing a method in wh liquids are contained in a vertical capillary tube attached t chamber The value of the interfacial tension may from measurements of the pressure required to drive up the capillary tube until the surface of the liquid em the upper end of the capillary is exactly plane The m promising, but I have not as yet any figures to quote

Research along some such lines as these would do muc trustworthy data for the solution of many of the problen chemistry and physics

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REPORT ON COLLAGEN AND GELATIN

By Professor HENRY RICHARDSON PROCTER (*University of Leeds*)
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Owing to the wide separation of the joint-authors of the report, and the impossibility of personal co-operation, it has been found necessary to divide the work, and J A Wilson takes the responsibility for the summary of American progress, and H R Procter for that of European With regard to the latter, its author must apologise for some incompleteness, as circumstances have compelled its completion in the absence both of his own, and of general scientific libraries, but it is hoped that it may prove sufficient as a general summary of progress made since the last report Attention has been drawn by footnotes to cases in which the two summaries are mutually confirmatory, or in which they may appear to differ

1 *American Work*

There is still some dispute as to the chemical relationship between collagen and gelatin, particularly as to whether collagen is an anhydride or merely a dehydrated form of gelatin Collagen is converted into gelatin by heating in water at 70° C Bogue finds that heating dry gelatin for 15 hours at 110° C renders it insoluble, but that reheating the insoluble mass in water at 70° again renders it soluble He accepts this as supporting Hofmeister's view that gelatin is converted into its anhydride, collagen, by heating [illegible] as a highly dehydrated gelatin converted into a [illegible]e approach of its constituent particles, rath[illegible] of gelatin C R Smith found that gelatin dried at 100° C and then heated to

128° C loses 1 25 per cent in weight It then swells very slowly and dissolves in water at 35° C to 40° C , with nearly complete restoration of its jellying power He concedes that gelatin dried at 128° C may be converted into collagen, but considers that collagen itself may represent a form of gelatin which is difficult to disperse Wilson and Daub found no visible tendency for the collagen fibres of calf skin to dissolve during 24 hours' contact with water at 40° C , even in the presence of pancreatin and at *p*H values ranging from 4 to 10 This fact makes it appear doubtful that the product which Smith obtained by heating gelatin to 128° C was identical with the collagen of calf skin Plimmer says that " those proteins which are resistant to the action of trypsin until they have been acted upon by pepsin will have all their units contained in the anhydride ring " If true, this statement would lend support to the view that collagen is an anhydride of gelatin, since trypsin will hydrolyse gelatin, but not collagen unless it has previously been attacked by pepsin

Gelatin is hydrolysed by both pepsin and trypsin Northrop, in making a comparison of the relative velocity of hydrolysis of the various peptide linkings of the gelatin molecule when hydrolysed by acid, alkali, pepsin and trypsin, found that those linkages which are most rapidly attacked by the enzymes are split most easily by alkali and are most resistant to acid hydrolysis Similar studies upon collagen have apparently not yet been made

Michaelis and Grineff found the isoelectric point of gelatin to lie at $pH = 4\ 6$ In a great variety of experiments, Loeb confirmed this value approximately, finding 4 7 But only very recently has any direct effort been made to determine the isoelectric point of collagen Working with standard hide powder, Thomas and Kelly obtained the value of 5 by means of experiments with acid and basic dyestuffs, while Porter found 4 8 by swelling experiments, indicating that collagen and gelatin have practically the same isoelectric point But in their experiments upon bating, Wilson and Daub found the minimum plumping of calf skin to occur in the region of 6 1 to 9 8[1], but it is recognised that a skin contains several proteins and it is even possible that the collagen of calf skin is not identical with that of the hides used for making hide powder , in fact, calf skin seems to yield a higher grade of gelatin than heavy hides Their test, however, was not sufficiently delicate to place the isoelectric point exactly

Loeb uses the isoelectric point as a means of purifying gelatin and other proteins, reasoning that the protein will combine with neither anions nor cations at its isoelectric point and that it can therefore be freed from impurities by bringing it to its isoelectric point and then washing The general procedure for gelatin is to wash it with successive portions of 0 002 M hydrochloric acid below 20° C , and then with

[1] Porter (*J S L T C* , 1921, p 281) in experiments in the Procter Research Laboratory, found the point of minimum swelling of sieved hide-powder to be very sharply at *p*H = 4 8, with a short rapid rise, and then very slow increase from *p*H = 6 to *p*H = 10 The minimum swelling of Wilson and Daub seems to cover this almost flat part of the curve, and unless they used buffers at very short intervals between *p*H = 4·5 and 6 0, they might easily miss the actual minimum altogether

distilled water to remove the excess of acid Field used this method and showed that gelatin so treated is ash free and at its isoelectric point The purified jelly obtained was opaque and white, but became transparent upon the addition of either acid or alkali Smith, apparently unaware of Loeb's earlier work, suggested a similar procedure, but found that the washing could be facilitated by treating first with salt water, to prevent swelling until the acid was removed, and then with distilled water to remove the salt

Experiments upon the mutarotation of gelatin led Smith to suggest that gelatin itself exists in two forms a sol form stable above 35° C and a gel form stable below 15° C , a condition of equilibrium existing between the two forms at intermediate temperatures At 15° C or less, the specific rotation of gelatin is practically constant at $(a)_d = -313$ for all concentrations, while at 35° C or over it is practically constant at $(a)_d = -141$ for all concentrations But at intermediate temperatures it increases with the concentration Moreover, gelation does not take place at any concentration above 35° C These facts are taken to indicate the existence of two forms of gelatin, the gel form having a specific rotation of $(a)_d = -313$ and the sol form a specific rotation of $(a)_d = -141$ Measurements of the velocity of mutarotation lead to the view that the change from the sol to the gel form is a reversible, bimolecular reaction, two molecules of the sol form uniting to give one molecule of the gel form Increase in lævorotation parallels increase in viscosity, due to the increasing proportion of the gel form He calculates that a maximum concentration of from 0 6 to 1 0 gram of the gel form is required to produce gelation As the temperature is increased, the total concentration of gelatin required to produce gelation is increased because of the decreasing proportion of the gel form, which does not exist at all above 35° C Gelatin is the only protein known to show mutarotation, but it gradually loses this property along with its jellying power, when its solutions are kept at temperatures above 70° C

Alcohol precipitates gelatin from solution, but this action is hindered by the presence of electrolytes Fenn found, with increasing concentration of strong acids or alkalies, that the retarding effect passes through a sharp maximum, at which no precipitation occurs at any concentration of alcohol, and then decreases to a constant level Salts which hydrolyse readily, like aluminium chloride, act much like strong acids in this respect The effectiveness of strong acids and alkalies in preventing precipitation is reduced by the addition of univalent salts Polyvalent cations are much more effective than univalent ions in reducing the effect of alkalies and polyvalent anions likewise in reducing the effects of acids in preventing precipitation by alcohol But when polyvalent cations and anions are present together, they reduce each other's effects [2]

The effect of electrolytes in hindering the precipitation of gelatin by alcohol is paralleled, within limits, by their effect upon the swelling

[2] Procter (*Koll Beihefte*, 1911, 2, 270) found that jellies swollen by acids could not be dehydrated by absolute alcohol, but that the addition of acid to the alcohol increased its dehydrating effect

of gelatin jellies and the osmotic pressure, viscosity and other properties of gelatin solutions Certain schools of chemists have assumed that these effects are not governed by the simple, quantitative laws of chemistry and have sought to explain them in terms of "hydration capacity," "Hofmeister series," "lyotrope influence," and other vague terms, which upon careful examination prove to offer no explanation at all Loeb and Wilson in America, however, have been staunch supporters of the view put forth by Procter that the properties of gelatin in the presence of electrolytes can be explained quantitatively in terms of orthodox physical chemistry, and this view now seems indisputably established In the third report Wilson showed that Procter's data could be calculated with great accuracy from the simple assumption that gelatin combines chemically with acids or bases, forming highly ionisable salts Since that report was prepared, Loeb has accumulated a great mass of data further corroborating this view

He showed clearly that the Hofmeister series is not the correct expression of the relative effect of ions on the swelling of gelatin, and that it is not true that chlorides, bromides and nitrates have "hydrating" and acetates, tartrates, citrates and phosphates "dehydrating" effects At the same *p*H value, the chloride, nitrate, trichloracetate, tartrate, succinate, oxalate, citrate and phosphate of gelatin swell to the same degree, while the swelling is considerably less for gelatin sulphate, which is exactly what would be expected on the basis of the combining ratios of the corresponding acids The anions of the weak polybasic acids are almost entirely monovalent, whereas the anion of the strong sulphuric acid is divalent It is the valency rather than the nature of the anion that affects the degree of swelling The divalent anion has no greater osmotic pressure than a monovalent one, but must distribute it between two protein equivalents and we get therefore only about half the degree of swelling This is further corroborated in experiments with alkalies, the hydroxides of lithium, sodium, potassium, and ammonium cause the same degree of swelling at the same *p*H value, whereas calcium and barium hydroxides cause only about half of this degree of swelling The ions also act in a similar sense upon the relative solubility of gelatin in alcohol-water mixtures Conductivity measurements show that the drop in curves for swelling, osmotic pressure, or viscosity at *p*H values decreasing from 3 is not due to repression of the ionisation of the gelatin salts and that the difference in properties of gelatin sulphate and chloride is not due to differences in degree of ionisation of the two salts

Loeb's experiments seem to indicate that proteins combine only with cations on the alkaline side of the isoelectric point and only with anions on the acid side According to the Procter-Wilson theory, the combination of protein and hydrogen ion is governed by the law of mass action, which means that theoretically there must be some combination at any *p*H value However, a calculation from this theory shows that at 4 7 only 0 4 per cent of the gelatin would exist as cation, while at a *p*H value of 6 this would be reduced to a few thousandths of 1 per cent Loeb's experiments are, therefore, fully

in keeping with the theory At the isoelectric point the 0 4 per cent of gelatin cation must, of course, be balanced against an equivalent amount of gelatin anion [3]

Aminoacetic acid furnishes an example of a molecule that can have one group acting as anion and another as cation simultaneously, the positive charge at one end of the molecule being balanced by the negative charge at the other Chromium ion will enter into stable combination with collagen even at *p*H values as low as 2 5, which is well over on the acid side of the isoelectric point The amount of collagen anion existing at *p*H = 2 5 must be extremely small, although not so small as that yielded by chromium collagenate The rate of combination of chromium and collagen decreases with decreasing *p*H value, as we should expect because of the decreasing proportion of collagen anion

In his earlier experiments, Loeb gained much valuable information regarding the chemical properties of the proteins by treating them first with an electrolyte of definite concentration and then washing away the material not combined with the protein Although this procedure is open to the criticism that the washing disturbs the equilibrium and therefore does not give absolutely accurate results, it has nevertheless given much valuable information over a limited range of *p*H values on either side of the isoelectric point By this method he found that the amount of ion combined with the protein is proportional to the osmotic pressure, viscosity and swelling of gelatin, and that twice as many univalent as bivalent ions combine with the same weight of gelatin at the same *p*H value He was thus enabled to show that the combination of gelatin and electrolytes follows stoichiometrical laws At very high or very low *p*H values, when the excess of electrolyte is not washed away, it causes a decrease in osmotic pressure, viscosity and swelling, but with no reduction in the amount of ion combined with the gelatin

A most important development of Loeb's work was the establishment by experimental proof of the view long maintained by Procter that Donnan's work on membrane potentials applies quantitatively to gelatin-electrolyte equilibria The formula for this as given by Procter and Wilson is

$$E = \frac{RT}{F} \log \frac{x}{y} = \frac{RT}{F} \log \frac{2x}{-z + \sqrt{4x^2 + z^2}}$$

where E is the difference of potential between the external solution and the jelly phase, x the concentration of electrolyte in the external solution, y its concentration in the jelly, and z the concentration of gelatin ion They pointed out that E must increase to a maximum and then decrease approaching zero as x increases without limit

[3] While the combination with acids or bases at the isoelectric point in dilute solutions is small or nil, it seems probable that in more concentrated solutions of neutral salts, the gelatin may combine in appreciable quantity with both the anion and the cation of the salt Procter (*Koll Beihefte*, 1911, 2, 270) observed that neutral gelatin jelly absorbed a considerable quantity of sodium chloride positively, though as soon as acid was present and gelatin chloride formed the absorption immediately became negative

It will be evident from the published work of Procter and Wilson that, regardless of the number of kinds of ionogens present in the system, the ratio of concentration in the jelly phase to concentration in the external solution must have the same value for all ions of one sign and the reciprocal of this value for all ions of the opposite sign The potential difference caused by the unequal distribution of any one ion between the two phases is the same as that caused by the unequal distribution of all others By measuring the pH value of both the jelly and the solution, Loeb was thus enabled to calculate the potential difference, since

$$E = \frac{RT}{F} \log \frac{[H^+]_{soln}}{[H^+]_{jelly}} = \frac{RT}{F} (p H_{jelly} - p H_{soln})$$

His next step was to determine this value experimentally, which he did both for blocks of jelly immersed in their equilibrium solutions and for solutions of gelatin separated from their equilibrium solutions by means of collodion membranes The general procedure was to take two calomel electrodes of equal value, dip the tube end of one in the solution and imbed the end of the other in the jelly, while jelly and solution were in contact and at equilibrium, and then to measure the voltage of this system by means of a Compton electrometer An example of the remarkable agreement obtained for the effect of sodium nitrate in depressing the potential difference is given in the following table —

TABLE I

Concentration of Sodium Nitrate	(Millivolts) Potential Difference Calculated	Observed
0	31 2	31
M/4096	28 3	28
M/2048	24 0	24
M/1024	20 7	22
M/512	16 0	16
M/256	11 2	12
M/128	7 0	7
M/64	4 1	4
M/32	0 6	0

(Original inside solution, 1 per cent originally isoelectric gelatin dissolved in various concentrations of $NaNO_3$ made up with HCl to pH = 3 5 Outside solutions, same concentrations of $NaNO_3$ all made with HCl to pH = 3 0 pH values determined after 18 hours)

The Procter-Wilson theory of vegetable tanning puts these membrane potentials to practical use by pointing out that the apparent astringency of a tan liquor is measured by the sum of two potential differences, that existing between the jelly phase of the collagen fibres and the tan liquor and that existing between the tan liquor and the thin film of solution surrounding each tannin particle. Thomas and Foster have since actually demonstrated that those tanning extracts which are most astringent under fixed conditions have the greatest potential difference between the solution and the surface film of the particles,

Because of its insolubility, collagen does not lend itself so readily to experiment as gelatin, but experiments on the latter often furnish information concerning collagen by analogy Taking the combining weight of gelatin as 768, Wilson suggested that this differs from the combining weight of collagen by the weight of one molecule of water, making the latter 750 He calculated from this that the minimum amount of chromic oxide required to produce chromium collagenate, or chrome leather, is 3 38 grams per 100 grams of collagen In studying the effect of concentration of a chrome liquor upon absorption by hide substances, Baldwin obtained a maximum fixation of 13 4 grams of chromic oxide per 100 grams of hide protein and referred to this as tetrachrome collagen, since it was almost exactly four times the minimum value calculated by Wilson Later Thomas and Kelly confirmed Baldwin's work and then extended it to a study of the results of 9 months' contact of hide protein and chrome liquor They then obtained a maximum fixation of 26 6 grams of chromic oxide per 100 grams of hide protein, just eight times the calculated minimum, and this they called octachrome collagen Moreover, in the curve plotted for the amount of chromic oxide fixed as a function of the concentration of the liquor, they found a point of inflection exactly at the point corresponding to tetrachrome collagen

Uncertainty as to the exact composition of the tannins makes similar calculations in the field of vegetable tanning much more highly speculative Taking one-fifth of the molecular weight of penta-digalloyl glucose as its equivalent weight, Wilson calculated 45 3 grams of tannin as the minimum required to form collagen tannate In tanning calf skins on a large scale, this actually proved to be very close to the minimum proportion of fixed tannin to hide protein required for the leather to pass as completely tanned, while just double this proportion appeared to be the maximum possible under the conditions of the tan yard This work has not yet been accurately corroborated by laboratory experiments

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2 *European Work*

The equilibrium of gelatin and collagen with acids and alkalies has continued to receive attention Perhaps the most important paper recently published is one by Miss D L Lloyd[1] (This paper has been reprinted in full, with a criticism by the Editor, in the *Jour Soc Lea Trades Chemists*, IV, No 7, 1920 and a few copies can be obtained from the Associate Editor, Leeds University, price 2*s*) Miss Lloyd experimented with discs of Coignet's "Gold Label" gelatin, containing 20 per cent of moisture and 1 0 per cent of ash, which were exposed to the solutions in Gooch crucibles, in which they were subsequently weighed after rapidly wiping with filter paper The acidity of the solutions was ascertained, both before and after the experiment by electrometric measurement, and the swelling was in a thermostat at 20° C, a few drops of toluene being added to prevent bacterial action. 150 c c of solution were usually employed, and this in some cases proved insufficient to keep the pH above or below the isoelectric point when equilibrium was established Time-curves were obtained, extending in some cases to 11 days which tended to become horizontal up to about 5 days, but with solutions of HCl of 0 05N and upwards, increased swelling then occurred, leading ultimately to solution A similar result occurred with NaOH solutions of 0 005N and over, and with concentrations exceeding this hydrolysis was obvious almost from the commencement In acid solutions a maximum swelling occurred at about pH = 2 4, dropping to a minimum at the isoelectric point, and rising more gradually on the alkaline side to a maximum about pH = 11 8 with a subsequent drop and second rise before complete hydrolysis This drop and second rise has been shown by Porter to be due partly to progressive hydrolysis, and partly to the repressive effect of the increasing OH concentration

Miss Lloyd found no evidence of molecular breakdown in solutions either in HCl or in NaOH if these were examined *immediately on solution*, but while the gelatin could be recovered, apparently unaltered, by neutralisation and precipitation with alcohol or ammonium sulphate, that from acid solution was still capable of gelatinisation, while the alkaline refused to gelatinise, and must have suffered some molecular change

Miss Lloyd makes some interesting remarks on Paul von Schroeder's observation that the maximum swelling of gelatin differs in water and water-vapour, and points out that though this is probably not true of isoelectric gelatin, contraction must occur where the jelly has been in Donnan equilibrium with an acid or alkaline solution, when by removal into vapour the osmotic pressure of the external solution is lessened or removed Procter and Wilson have shown that for a gelatin jelly to be in thermodynamic equilibrium with an external acid solution it must have an excess of osmotic pressure over the latter which they have denoted by e, and which must be balanced by the internal attractions of the jelly which Miss Lloyd has described as its "recoil" When the external solution is removed, this pressure e is no longer balanced, and a portion of the internal acid liquor is forced out, which is more dilute than the original external acid, and with which the jelly ultimately comes into equilibrium This pressure e is apparently directly proportional to the swelling, which therefore obeys Hooke's law that the elastic stress is proportionate to the strain, and therefore to the increased volume This law is the same as would result from surface energy if the internal surface were proportionate to the volume, and, like the surface tension, is no doubt connected directly with the internal pressure of the jelly

Miss Lloyd's paper must be characterised as a most useful piece of experimental work, though her theoretical conclusions are not in all respects confirmed by present knowledge W R Atkin[2] shows that her measurements agree very closely with those of Procter, Procter and Wilson, and Loeb, and with those which can be deduced from the equations of the Procter-Wilson theory

A further paper by W R Atkin[3] describes more recent work of Loeb's on acid treatment of powdered gelatin, and its effects on swelling, viscosity and osmotic pressure, and points out their concordance with those of Procter and his collaborators, and with the Procter-Wilson theory of swelling The actual hydrion concentration is shown to be the factor determining swelling, irrespective of the nature of the acid, and its place in the so-called "lyotrope series" The volume of swelling is practically the same with all monobasic acids, and with the weaker dibasic, where the ionisation of the second displaceable H is so small as to be negligible, but with sulphuric, and probably with other "strong" dibasic acids, the maximum swelling, though it occurs at the same pH (2 4 for the internal acid, corresponding to about 3 0 of the external), is only to about half the volume of that with monobasic acids, since the osmotic pressure of gelatin sulphate is only half that of gelatin chloride Loeb has shown that the same consideration applies to the gelatinates, the compounds with diacid bases, such as lime and baryta, having only half the osmotic pressure, and swelling to only half the volume of the monacid ones such as potash and soda

In another paper on the "Chemistry of Lime Liquors," W R Atkin[4] has dealt with the alkaline swelling of hide (collagen) in more detail, showing the practical effects of monovalent and divalent bases, swelling in lime liquors being increased by the former, even in the

form of neutral salts, and the smoothness of grain obtained in lime liquors strengthened by arsenic sulphide being simply due to the absence of sodium salts Alkaline swelling is shown to be exactly analogous to acid swelling, and an effect similar to "pickling" can be produced by large excess of the alkaline salt

Reference must be made here to some work of Loeb s[5] on viscosity, which has not been specifically made by Wilson, though the papers are mentioned in his bibliography He gives conclusive reasons for believing that the high viscosity of proteid solutions is not due to the hydration of their individual ions or molecules, but to the presence of particles of proteid jelly which absorb liquid in accordance with the Donnan equilibrium, and that the viscosity increases with their size in approximate concordance with the Einstein formula $\eta = \eta_0 (1+2\ 5\rho)$ where η_0 is the viscosity of the pure solvent at the temperature of experiment, η the viscosity of the solution, and ρ the fraction of its volume occupied by the solute In confirmation of this view he determined the viscosity of suspensions of finely powdered gelatin in solutions of varying pH, and found it materially greater than that of actual solutions of gelatin of the same concentration, and in accordance with the known volume of the particles of the swollen gelatin as affected by the Donnan equilibrium The connection between the swelling and the increase of viscosity is therefore a clear one With lowered temperature, and with increase of time after actual solution, the size and number of these particles increases, and with these the viscosity Loeb appears to think this explanation sufficient to account for the observed viscosities, but his experiments were made with relatively concentrated solutions and at temperatures above or near the setting point, and reasons have been given for believing that even with solutions so dilute as to remain liquid, below this temperature, a viscosity of structure also occurs

Much work has been devoted to elucidation of the ultimate structure of jellies in general, and especially of gelatin jelly, and some of the views held, which are apparently opposed, are probably not so in reality Since it has been shown by Hatschek[6] that the emulsion theory of two liquid phases with interfacial tension is incapable of mathematically explaining the elasticity and other observed properties of jellies, it has been largely abandoned in England, though it is still held by some German chemists, and especially by the Ostwalds, and the view seeems to be gaining ground that the solidification of a jelly is a process allied to crystallisation, and that its ultimate structure is that of a network of crystalline fibrils of little more than molecular dimensions, separated by liquid This view is not in any way opposed to that advocated by Procter and Wilson, who only claim that a jelly is homogeneous in the sense that all its parts are within the sphere of molecular forces, and can be treated as a single phase in equilibrium with an external solution This does not at all preclude the idea of a further equilibrium, within the phase, between the [illegible] and the liquid portion It has been pointed out by [illegible] expulsion of liquids from jellies does not involve the existence of two phases in the sense of the phase-rule, but is a

necessary consequence of the ordinary laws of osmotic equilibrium, and only implies the mechanical separation of two phases in the same sense as the mechanical separation of salts from their solutions by powerful centrifugation Bradford, Barratt, McBain and many others only seem to differ in their ideas as to the precise form of crystallisation, and they are supported in their view of crystalline structure by many of the most eminent Continental authorities, such as von Weimarn, Zsigmondy, and others It is well known that many definitely crystalline substances, such as benzopurpurine, chrysophenine and cholic acid, set from concentrated aqueous solutions into substances with all the properties of jellies, though the crystals ultimately grow to visible size W Harrison[8] mentions that crystals of cholic acid precipitated with iodine, though sometimes rigid, may be so tenuous as to be quite flexible, and may be twisted into spirals by the bombardment of liquid molecules Camphorylphenylthiosemicarbazide prepared by M O Forster[9] forms stiff jellies at concentrations so low as 0 33 per cent McBain has shown that soap curds consist invariably of fine fibres, which in the case of sodium soaps may be many centimetres long, but never greater in diameter than 1μ[10] The actual jellies are very transparent, and their structure does not seem to have been determined, but presumably it is still more minute

Some interesting observations on the growth of collagen fibres in healing wounds have been made by W von Gaza, a German army doctor[11] He points out that the connective tissues are very permanent, and consequently in the adult sparingly provided with formative cells, which do not form part of the fibres, but exist between them, though in embryonic development the cells are much more abundant The connective tissue fibrils are only formed by these cells, and are at first very slender, though whether the fibril, once formed, can grow in thickness at the expense of the surrounding paraplastic matter the author leaves undecided In a wound, the connective tissue becomes swollen, softened and dissolved, though no appreciable quantity of gelatin appears to be produced, and the author is of opinion that a colloidal, rather than a chemical change takes place, while this softening is necessary to allow of the welding together of the broken tissues At the same time a great increase in the number and activity of the connective tissue cells takes place, and they rapidly divide and increase as in the embryo The tissue when first formed is exceedingly soft and tender, but afterwards contracts and hardens, though for three weeks it can bear very little mechanical stress

The double refractive effects frequently shown by connective tissue (collagen) fibres under polarised light suggest crystalline structure, but are probably merely due to mechanical strains Such effects are also shown by gelatin under stress, either mechanical or produced by unequal drying, and Hatschek[12] has shown that these strains persist after the stress is removed, while Harrison has observed similar phenomena with other colloid fibres It has, however, been shown by H Ambronn[13] that if fine rods, parallel to each other, are embedded in some medium of different refraction so as to form a

grating, the system behaves like a uniaxial positive crystal In a medium of similar refractive index this effect disappears, while true double refraction persists Cellulose fibres produce such effects, together with a strong double refraction which does not disappear in media of the same index, but is reversed in sign on nitration, and returns to its original form on denitration, clearly indicating its chemical nature

As regards the form of crystallisation which causes gelatinisation, there is considerable difference of opinion, and as the crystals are, at least in their beginning, amicroscopic, it is impossible directly to observe their form, but probability seems to point to their being molecular chains It may be remarked that most observations have been made on neutral or isoelectric gelatin, which from its extreme insolubility in water is perhaps most easily obtained in solid form, but as acid gelatin sets to stiff jellies, and the compounds are evidently definite salts, it seems desirable that efforts should be made to crystallise them

Bradford[14] experimenting on neutral gelatin, found that a 0 4 per cent solution sterilised and sealed in clean test-tubes, became opalescent after three weeks, and the opalescence settled down in the tubes at a rate corresponding to an average diameter of 0 1μ Six weeks later, the appearance was that of innumerable grains just too small to be distinguished, but a fair number of larger grains were visible, up to 0 6μ in diameter Some of the spherical grains in these experiments appeared to dipolarise light Von Weimarn's formula shows that by working at a higher temperature, larger grains should be produced, and by working at 31° C and 40° C this was found to be the case, and on cooling well-developed spherites, up to 1μ in diameter were obtained, some of which showed a glowing central point with crossed nicols In course of time the gels tended to contract and shrink away from the sides of the tubes Bradford apparently views jellies as crystalline structures, containing supersaturated solution

Barratt[15] takes a very similar view to Bradford, and inclines to the idea of fibrillar crystals, generally in tension (and consequently straight) He points out that in [illegible] the first fibrillar structure which can be shown to [illegible] and therefore invisible, but that the fibrils grow by increasing in thickness This hypothesis not only affords a plausible explanation of the observed properties of jellies, but also accounts for their behaviour in ultrafiltration[16]

Hatschek[17] remarks that McBain is no doubt right in differentiating sharply between curds and gels, but that no state similar to the soap-curds exists in gelatin jellies If, however, alcohol is added to a neutral gelatin solution, or salt to an acid one, the liquid first becomes turbid, then separates into flocks, and finally to masses of more or less dehydrated jelly, which rapidly agglutinate It is possible that no difference in principle exists between the soap-curds and these gels, except that the former, from the conditions of the solution, parts more completely with its surrounding liquid, and shows greater

tendency to agglutination The latter property must be largely dependent on the interfacial tension between the colloidal particles and the surrounding solution, and as the surface-charge of the latter is diminished by the addition of alcohol or salt, the interfacial tension must corresponding increase It is highly probable that the phenomena of " salting out " are due to the fact that while in concentrated salt solutions, the Donnan potential becomes practically zero, the surface-tension correspondingly increases, and causes agglutination

It is certain that jelly-structure exists in gelatin solutions below the setting temperature, even when too dilute to show any visible signs of gelatinisation This is shown, not only by the rapid increase of viscosity near the setting point on cooling, but by the fact that a constantly increasing portion of this viscosity is due to a structure which can be mechanically broken, and which shows elasticity It is well known that such solutions, when treated in the ordinary capillary viscosimeters show a diminution of viscosity on being passed through a second time, and that their viscosity is materially diminished by violent shaking When on the other hand elasticity is measured by the torsion required to twist an immersed cylinder, it is found that up to a certain degree of movement the cylinder will return to or towards its original position on the stress being relaxed, an action which is not observed in liquids which owe their viscosity merely to internal friction The Kundt [18] phenomenon of dipolarisation observed in such liquids when submitted to rotational stresses (*vide supra*) is also evidence of structure On this view, Arisz's observation of increased viscosity and Tyndall effect on cooling become comprehensible since while jellies in equilibrium with an external solution are optically and mechanically homogeneous, their diluted solutions probably consist of colloid particles in equilibrium with the surrounding solution, which increase in size and become more concentrated as their internal cohesion increases by cooling At and over 70° C gelatin solutions are probably approximately molecular, but below this become increasingly colloidal There is good reason to believe that the same equilibrium as in massive jellies exists between the gelatin-particles and their surrounding liquid phase On cooling, even dilute solutions set to a coherent jelly, but there seems some evidence of their tendency to contract and separate liquid till the concentration of the jelly becomes such as is required for equilibrium with its external solution

Much speculation has been indulged in, both in England and on the Continent as to the cause of the Liesegang effect, and some interesting experiments have been made with other salts than silver nitrate and potassium dichromate In its original and simplest form the experiment consisted in placing a minute crystal of dichromate on a wet gelatin film containing a little silver nitrate, when the diffusing chromic acid produced a series of successive rings of silver chromate in the gelatin, becoming closer and fainter as their diameter increased A similar phenomenon is the production of successive layers when dichromate solution is allowed to diffuse downwards in a tube of gelatin jelly containing a soluble silver salt It has been shown that

Such effects may be produced by many (if not by any) pairs of salts which give together an insoluble precipitate Harrison[19] by diffusing copper sulphate into a gelatin jelly containing barium ferrocyanide, obtained alternate rings of copper ferrocyanide and barium sulphate, and with silver sulphate and barium chloride, alternate rings of silver chloride and barium sulphate Neither gelatin nor any other colloid seems essential to the production of these effects, and colloids probably act only in slowing diffusion, and preventing convection currents J Traube[20] obtained similar effects by allowing ferric salts to diffuse into plaster of Paris containing ferrocyanide, and many years ago agate-like structures were obtained by Ianson by allowing metallic salts to diffuse into water-glass, the writer believes his results were published in the Transactions of the now long extinct Newcastle Chemical Society

Similar results were obtained by H N Holmes,[21] who mentions that Chapin obtained band-effects in a glass tube 1 m long, into which HCl and NH_3 gases were allowed to diffuse from opposite ends Most of these results can be quite simply explained by the ordinary laws of diffusion If we imagine a dichromate solution diffusing into a jelly containing silver nitrate, a ring of precipitated silver chromate will be formed which will be practically devoid of either silver or chromic ions, and these will naturally diffuse into it from both sides continuing to be precipitated as silver chromate, until an outer zone is formed devoid of silver ions, into which further diffusing chromic ions can produce no precipitate till they reach a region where silver ions still exist, when the process will repeat itself Harrison's observation of alternate rings of silver chloride and barium sulphate is less easy of explanation, but may perhaps depend on differences in the rate of diffusion of the different pairs of ions

W Moeller[22] has several long papers on the rhythmical diffusion of hydrochloric acid in salted jellies He found that if a minute drop of concentrated hydrochloric acid were placed on a film of salted gelatin, a series of rings similar to those of Liesegang were produced, and he endeavours to prove that these are dependent on a pre-existing system of spiral fibrils in the jelly, a theory for which there seems no other evidence

Allusion must be made to the important work of Anderson[23] and continued by Bachmann[24] on the fine structure of jellies Anderson found that the water in silica jellies could be substituted by alcohol or benzol, and found that by applying the known formula

$$r = \frac{2\,T\,S_0\,\,0\,4343}{D\,\,p_0\,\,\log p_1/p_0}$$

where r = radius of capillaries, T = surface tension of liquid, S_0 = density of saturated vapour of liquid under ordinary conditions, p_0 = ordinary vapour-pressure of liquid, and p_1 that of the liquid imbibed, and D its density, the same value for r (about $5\mu\mu$) was found in each case Bachmann found that though aqueous gelatin jellies are contracted by strong alcohol, and will not absorb it, it was possible by gradually increasing the alcoholic strength, to substitute alcohol

for water without contraction of more than 50 per cent of its aqueous volume, and then in a similar way, to substitute benzol for alcohol. The jellies became hardened, and were no longer elastic, but still remained tough and transparent. Graphs are given for the somewhat complicated curves of vapour pressure, for which the original paper must be consulted, but which bear a general resemblance to those of van Bemmelen for silica jellies, generally showing a different curve of re-saturation with vapour to that of drying. The 30 per cent jelly at the zero-point gave an approximate capillary radius of about 6$\mu\mu$, thus nearly agreeing with that found by Anderson for silica jellies. The author concludes —

1. That the capillary measurements are unaffected by the nature of the liquid.
2. That they are also largely independent of the material of the gel.
3. That the pores are of a much finer character than Bütschli imagined, since his gross microscopic pores could have no appreciable effect on vapour-pressure.

While the paper is a very interesting one, and deserves careful study, it does not at all conclusively prove that the structure of really elastic jellies is of the same character as that of gels either naturally rigid, or made so artificially by hardening agents such as alcohol, and which have lost the power of swelling and contraction which is so characteristic of the natural jellies. The formula is only applicable to rigid gels, and while it is interesting that the numerical results with the aqueous jelly are so similar, it is perhaps not surprising, since the dimensions of the pores are almost molecular, and well within the range of molecular attractions.

Collagen.—There has been but little direct work on collagen, and it is not easy to see in what direction progress can be made, since there is no known means of bringing it into solution without altering its constitution, and the impossibility of obtaining it as a homogeneous mass makes physical experiment very difficult.

Apart from the determinations of iso-electric point and minimum swelling which have been already referred to (p. 14), and which do not seem to differ materially from those of gelatin, perhaps the most important work is that of V. Kubelka,[25] who made a long series of very careful experiments on the absorption of hydrochloric acid by hide-powder, using both an old and a newer sample of unchromed Freiberg hide-powder, which gave identical results within the limits of experimental error. About 5 gms. of powder and 100 c.c. of diluted acid were used for each experiment (though the volume of acid is not definitely stated), and the powder was shaken for one hour with the acid, as it was found that practical equilibrium was obtained in five minutes, and no further absorption took place in six hours. X/m was calculated by subtracting the acid found in 1 c.c. of the equilibrium-acid from that of the original concentration, multiplying by 100, and dividing by the weight of the hide-powder used, the titration being made with NaOH, using methyl-orange as an indicator. With an original concentration $y = 9\ 1284$, $X/m = 0\ 635$, and it

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COLLOID PHENOMENA IN BACTERIOLOGY

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The influence of the colloidal nature of micro-organisms on the processes of bacterial culture and in the operations of growth inhibition and of sterilisation is generally masked by the presence of factors which affect that delicate balance within the cell which is necessary for the operation of vital processes These have been detailed by Bayliss in the second report (pp 117–154)

Bacteria as colloidal systems—However diverse the action of growth stimulants, or however complicated the mechanism by which death is caused in the micro-organism, nevertheless in many cases, at least in the preliminary stages, the bacteria may be regarded as simple colloidal systems

The majority of the bacteria exhibit Brownian movement, possess an average diameter of 1 to 1 5μ, and a semi-permeable envelope of complex constitution, in which chitin, celluloses and fats may frequently be identified Within the envelope are contained salts such as sodium chloride, lipoids, polypeptides, carbohydrates and enzymes Microscopically they are to be regarded as suspensoid systems, but owing to the fact that the membrane is semi-permeable in many respects the micro-organisms resemble emulsoids rather than suspensoids In water, saline solution, and in many dilute nutrient media they acquire a negative charge and exhibit the phenomenon of electric cataphoresis They are only feebly precipitated by cations, and in accordance with the Hardy-Picton law the trivalent cations such as iron and aluminium are more effective than the di or monovalent ions, the latter indeed with the exception of hydrion in strong acid concentrations and the ions of the heavy metals exerting scarcely any precipitating effect Also, as in the case of the emulsoids, protective colloids such as isinglass and gelatine exert but little protection

against coagulation, although media rich in peptone have a distinct effect on the stability of the system [1]

Bacteriological research does not in general extend to those regions of instability brought about by heat coagulation, but interest is confined chiefly to the conditions of optimum growth or the effect of antiseptics and germicides on the growth rate Changes in the growth rate offer a much more sensitive criterion of environmental conditions than one can hope to attain by the relatively crude method of precipitation

Bacterial growth.—A study of the conditions of growth and of germicidal action has revealed the necessity of control of certain factors, due in part to the colloidal nature of the organisms In the preparation of nutrient media it is found necessary to maintain the P_H within certain limits for the particular species This is, in all probability, to be ascribed to the existence of some particular enzyme in the organism which possesses an optimum catalytic activity in the assimilative process within that range rather to its effect on the stability or the osmotic pressure within the organism, since very much higher acid concentrations than the nutrient broth range of P_H 8 to 6 are necessary completely to inhibit growth or to effect a noticeable germicidal action Auto-destruction by acid production during growth can be prevented by the utilisation of suitable buffer salts which exert but little inhibiting influence such as disodium hydrogen phosphate, sodium pyrophosphate[2], or mixtures of phosphoric and aspartic acids [3] In spite of the presence of the necessary food materials and control of the P_H auto-intoxication frequently occurs and evidently some product of bacterial growth exerts an influence on the growth rate The organisms cease growing On removal of the organisms by centrifuge the broth is still capable of providing the materials and conditions of growth for a new culture whilst the old culture will continue to grow in a new broth Quantitative data are lacking as to the growth rate under these conditions but a qualitative interpretation of the facts may be given in terms of the fundamental property of colloidal systems, viz., the existence of an extended adsorbing surface

For a long period the bacterial emulsion was considered to be in actuality a simple two-phase system, the medium and the cell contents

Any substance dissolved in the medium will if soluble distribute itself between these two phases, if in the same molecular state in each phase the distribution ratio will be $K = C_1/C_2$, where C_1 is the solubility in one phase, C_2 in the other, and will remain constant over a relatively wide range of concentration Thus Overton and Meyer[4] in their studies on narcosis laid special emphasis on the partition coefficient K = solubility in lipoid/solubility in water A high value for this coefficient would ensure a high concentration within the lipoid constituent of the cell relative to the environment Numerous investigations, such as those of Gossl[6] and Herzog and Betzel[5] have traced a very close connnection between antiseptic or disinfecting power and lipoid solubility Nevertheless, substances like the saturated paraffins possess scarcely any bactericidal activity although their lipoid/water partition coefficient is extremely high

Surface adsorption —The more recent developments in the study of colloidal systems have emphasised the important influence of the interfacial surface on colloidal reactions With bacteria presenting the extended surface of colloidal systems adsorption at the interface is a factor even more important than the lipoid water partition coefficient If any substance added to the medium lowers the interfacial surface tension then the surface concentration in accordance with the Gibbs's equation will be greater than the bulk concentration The relationship between the surface and bulk concentration may be expressed in the form

$$\Gamma = -\frac{C}{RT}\frac{dv}{dC}$$

where Γ is the surface concentration less the bulk concentration, *i e* the excess, C the bulk concentration, and v the surface tension, R the gas constant, T the absolute temperature It is evident that if the added solute has a great effect on the surface tension the surface concentrations may be quite high In some cases, *e g*, peptone and fuchsine, the surface concentrations for relatively dilute bulk concentrations may actually rise to values where precipitation of solid films takes place In the case of bacteria in a nutrient medium the surface concentration which is the concentration affecting the bacterium, may be much higher than the bulk concentration in the medium If during the growth of the organism substances are formed which tend to lower the interfacial surface tension in a marked manner, then, in accordance with the general theorem of mobile equilibrium of Le Chatelier, if the chemical processes involve the possible formation of such substances there will be a tendency for their production, and these substances will be adsorbed on the surface of the bacterium and affect the growth rate As the bulk concentration increases so does the surface concentration until the bacteria are no longer capable of growing If the quiescent bacteria are now removed by centrifuging they carry with them the adsorbed materials On seeding a new culture into the old medium clarified by centrifuging, growth can take place since the bulk concentration of the inimical materials left in solution is small and is readily reduced by the addition of the new bacteria Nevertheless the quantity of inimical substances to be produced before the new growth is paralysed is less than before and one would not expect the new culture to attain quite such a mature condition as the old Again if the old culture with its adsorbed materials be placed in a fresh medium the inimical substances will be partly desorbed until the Gibbs's equation conditions are re-established and growth will again ensue until the original inhibiting surface concentration be reached

Since no direct method is as yet available for the actual determination of the interfacial surface tension between bacterium and the medium we have to rely upon indirect evidence for support of the hypothesis That an adsorption does actually take place at any rate under certain specified conditions is evident from the work of Kuster and Bojakowsky[7] who showed that the partition of phenol between

water and anthrax spores followed the general empyric adsorption isotherm of Freundlich $x = ac^{\frac{1}{n}}$ where x is the amount adsorbed per gram of material, c the bulk concentration, a and n constants. Although it is not possible to measure the bacterium/medium interfacial tension yet the measurement of the medium/air interfacial tension is a comparatively simple operation If the assumption be made that most substances which affect the liquid/air interface will affect the membrane liquid interface in a similar manner, an assumption for which there is much evidence from chemical data, but which is by no means universally true , then it follows that many substances which depress the liquid/air interface will be strongly adsorbed by the micro-organisms and affect either favourably or unfavourably their growth rate This relationship between the lowering of the surface tension and germicidal activity is well exemplified in the somewhat insoluble hydroxyl derivatives

In order of decreasing molar germicidal activities we find thymol, camphor, menthol, the cresols, and lastly phenol These substances all depress the surface tension of water, thymol exhibiting a very marked effect, phenol being the least effective, the others lying in between these two in the above order Amongst the polyhydric benzene substitution products we again find this correlation between the surface tension depressant action and germicidal activity for, both these properties we find phenol > resorcin > hydroquinone > phloro-glucin > pyrogallol

A remarkable correlation is likewise found in the nitrophenols and nitrobenzaldehydes In the former the *p*-substituted products, in the latter the *o*-substituted products exert the greater molar germicidal activity , their action on the surface tension of water lies in this order Brown and Tinker[8] found a direct relationship between the adsorption of phenol by barley seeds and the surface tension More recently[9] it has been shown that the germicidal activity of phenol is raised by the addition of sodium chloride to the solution This elevation is accompanied by an increase in the fugacity of the phenol as measured by partition methods (*c f* G N Lewis[10]) Since sodium chloride elevates the surface tension of water its surface concentration must be less than its bulk concentration, the surface concentration of phenol must consequently increase on the addition of salt The germicidal activity of acids in the presence of neutral salts may be interpreted in a similar manner[11] The inhibiting materials produced during bacterial growth may be soaps, fatty acids and higher aliphatic alcohols Minute quantities of these materials affect the growth rate to a remarkable degree Thus 0 02 per cent of octyl alcohol[12] practically inhibits the growth of yeast (*See also* Toller and Clark and J H Wright[13]) In many micro-organisms especially in the yeasts growth is accompanied by fat formation which can undergo gradual hydrolysis or saponification in alkaline media The variability in " lemco " broth is generally ascribed to the presence of small traces of soaps formed during the process of manufacture The hydrocarbons possessing a high lipoid/water partition coefficient possess no germicidal activity, whilst the amount of chemical action between the hydrocarbon

and water as estimated by the magnitude of the independent and interfacial surface tensions is likewise inconsiderable

Attempts have been made by Larson, Cantwell and Hartzell[14] and others to determine the influence of the surface tension of the culture medium on the growth of bacteria and to grade nutrient broth and peptone waters by such methods Experiments in which the drop weight method is employed for such determinations are of but little value unless the rate of dropping is sufficiently slow to ensure the attainment of the Gibbs's equilibrium at the surface of the newly formed drop According to the writer's experience, five minutes per drop is by no means too long a time (*cf*, p 7) The static method is to be preferred for dilute solutions or for disperse systems such as the peptones, which possess small diffusion coefficients

No very marked alterations in the surface tension of a medium during bacterial growth is to be expected since the formation of surface tension depressants will be followed by their almost simultaneous adsorption by the bacteria, which on removal by centrifuging or filtration prior to the testing of the liquid will simultaneously withdraw the bulk of the depressing material

Chemical constitution and adsorption—For ionised substances the sign and magnitude of the electrical charge are important factors but not the only ones, the influence of the chemical nature of adsorbed ions being frequently marked, such as the fluoride and oxalate ions reacting with intra-cellular calcium ions, whilst the anions of the lower fatty acids are somewhat 'lipoid soluble The adsorption of organic substances from solutions by bacteria is to be ascribed to the presence of certain groups in the molecules The reverse of this, *i e*, the non-adsorption in the absence of these certain groups has been clearly demonstrated by the work on plasmolysis of plant cells by de Vries[15] and by Hamburger and Gryns[16] on blood corpuscles, as well as the later work of Overton and Lowe on plasmolysis through membranes which indicated that membrane permeability for organic compounds was associated with the absence of certain active groupings These active groupings in organic molecules are either acid or basic, *e g*, —OH, —COOH, $-NH_2$, although unsaturated groupings such as the double bond and $>CO$ and —CHO are not without influence Evidently the adsorption is chemical in its nature, a hypothesis utilised by Ehrlich in his work on the spirillocides and by Emil Fischer on the fermentation of the various sugars The relationship between chemical constitution and surface tension as developed by Rayleigh,[17], Hardy[18] and Langmuir[19] is therefore of importance The work of these investigators has clearly emphasised the chemical nature of surface tension The surface film consisting of a unimolecular layer of orientated molecules attached to the water surface by their polar groups apparently identical with the "active" groups of Hamburger and Lowe, the non-polar portions or the lipoid soluble part of the molecule resisting as far as possible immersion in the polar medium No great extension of this hypothesis is necessary to include adsorption by micro-organisms Lipoid solubility of part of the molecule adsorbed is desirable so that the molecule may attach itself to the lipoid constituent of the cell, but it

would appear that the metabolism of the organism is not interfered with by this process, *e g*, the feeble action of the saturated paraffins If, however, the molecule possesses active or polar groups, *e g*, —OH, $—NH_2$, —COOH, these react with similar groups but of opposite kind present on the membrane of the micro-organism and disturbance of growth occurs

We must, therefore, postulate on the cell membrane or within its pores at any rate in positions easy of access to the circumambient liquid the existence of active or polar groups, acceptors, which can react with any molecules present likewise possessing active groups It has been noted that these polar groups on the cell are either acidic or basic in nature It would appear that all micro-organisms possess both types of groups, *e g*, both $—NH_2$ and —COOH, *i e*, can react with both acidic and basic substances in solution but the power of reacting varies for each bacterial species, B coli, for example, will react to both acid and alkaline groups but is less resistant to basic germicides, its acid groups being predominant either in number or reactivity Staphylococcus conversely is more sensitive to acid germicides It is interesting to note that the former class of organisms are Gram negative and the latter Gram positive [20]

Selective action—The membrane surface possesses a definite structure and its surface molecules are consequently definitely orientated relatively one to another The presence of both acid and alkaline groups on the membrane has been demonstrated Hence to each molecule, building up the membrane surface possibly a complex polypeptide, we must ascribe one or more such active groups or acceptors to which the material undergoing absorption is attached To a sub-microscopic observer the adsorbing surface will consequently no longer appear uniform but consist of active groups both acid and basic, *e g*, $—NH_2$, —COOH, —OH, distributed over the surface in a perfectly regular manner giving the general appearance of a structure over a relatively wide area It has been shown that union with either the acid or basic acceptors leads to a general classification of germicides, in some cases coincident with the division observed in Gram staining Evidently this feeble exhibition of selective action of germicides dependent on unipoint contact is greatly augmented if the possibility of multi-point contact is envisaged If the germicide possesses more than one active group, *e g*, two $—NH_2$ groups in each molecule, these can evidently combine with two —COOH acceptor groups on the micro-organism provided that the distance apart between the two groups on the germicide is comparable to the distance apart of the acceptors on the micro-organism Since the bacteria will vary among themselves both in number, nature and pacing of the acceptor groups, a very selective action is to be expected provided the suitable reacting germicide can be found or synthesised In favour of this hypothesis may be mentioned the facts that the dyestuffs possessing more than one active grouping are more selective in their action than the ions or simple organic compounds (*Methylene blue*[21], *diphenyl and triphenyl-methane dyes*[22], *rosanilines*[23], *flavine*[24] *and trypan red* [25])

Again the combination of toxins and anti-toxins and the reactions between the various sugars and their particular enzymes are simply interpreted on this hypothesis of multipoint contact The hypothesis leads to an interesting speculation as to the possibility of enzyme synthesis by the insertion of the particular reactive groups at the proper distances apart in a hydrocarbon chain, by hydrolysis of the complex cell structure of the micro-organism which produces the enzyme, and by bridging over the gaps between the reactive groups on the membrane surface by chemical reaction Evidently the old simile of key and lock can readily be reinstated in terms of complicated multipoint contact produced by intermolecular reaction

Professor Bayliss has already in the second report on colloid chemistry discussed the conditions of stability of the protoplasm and cell contents in the section on colloid chemistry in physiology Bacteria react in manners similar to those there enumerated for simple cells, thus death may be occasioned by protein precipitation within the cell as brought about by phenol and the ions of the heavy metals, by peroxide reaction with the protein as in the case with p-quinone,[6] or by the Clowes's phase inversion produced by calcium and potassium soaps

In the preparation of disinfectants attempts are made to augment the selective adsorption of the germicide, thus the chloramines containing the —NHCl group and probably the iodamines —NHI are much more germicidal than the free halogens, the possession of the active —NH grouping augmenting the activity In the preparation of the emulsified disinfectants the germicidal power for a given tar-acid content is nearly proportional to the fineness of the emulsion The tar-acid emulsions are generally made with vegetable oils and soaps containing a little free alkali and frequently stabilised by means of some protective colloid such as gelatine Under the microscope a continuous bombardment of the bacteria by the fine emulsion particles is to be observed, one or two frequently adhering for comparatively long periods From an analogy with the protected colloidal metals it would appear disadvantageous to augment the stability of the emulsified disinfectants above that necessary to ensure stability against precipitation by electrolytes in the concentrations usually met with in bacteriological practice, since the rapidity of action is governed by the reaction rate between the two colloidal systems

Conclusions—In addition to the factors such as the presence of suitable food materials, optimum temperature, P_H and the like, the growth rate of micro-organisms is greatly influenced by the concentration at the bacterium surface of the various substances present The surface concentrations may already be considerable even when scarcely detectable amounts are present in the bulk of the medium The conditions necessary for favouring high surface concentrations are shown to depend on the action of the substance on the interfacial surface tension The magnitude of this action can in many cases be estimated by the effect of the solute on the air/liquid interface

Adsorption appears to be a chemical process, reaction occurring between reactive groups in the material adsorbed and the ones (acceptors)

in the micro-organism Single point reaction leads to a simple differentiation between acid and alkaline reactive groups whilst multi-point action increases the selective nature of the reaction which ultimately became entirely specific

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INDUSTRIAL APPLICATIONS OF WETTING POWER

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Many industrial processes are designedly based upon the power of a liquid to wet the surface of a solid, *e g*, mineral separation processes, whilst in many other industrial operations it is now recognised that wetting power plays an important part, *e g*, tree sprays and cattle dips

The ability of a liquid to wet a solid surface, *i e*, to give an even, continuous film over it, is dependent upon three surface tensions the surface tension liquid/air (T_1), the surface tension solid/air (T_2), and the interfacial tension liquid/solid (T_{12}) For the liquid to wet,

$$T_2 \text{ must be } > T_1 + T_{12}$$

Of these three surface tensions, two, viz, T_2 and T_{12} are indeterminate, so that direct experimental proof of the above relationship is not possible Rontgen,[2] however, has succeeded in demonstrating its validity for the system rubber/water Cooper and Nuttall[3], by using a surface covered with a thin layer of castor oil, the interfacial tension of which towards the wetting liquid could be determined, were able to confirm its validity under somewhat different conditions

In the wetting process, in addition to these three surface tensions, consideration must also be given to two other factors (1) wetting is sometimes prevented by the presence on the solid to be wetted of a thin coat of extraneous matter, *e g*, grease, etc, Freundlich[4] points out that mercury fails to wet the surface of many solids owing to its inability to dissolve the surface layer of moist air, adsorbed on them, (2) sometimes the wetting power of a liquid is higher than would be expected from a consideration of the above three surface tensions Such is the case with saponin, proteins and various other substances which exhibit the property of surface concentration (capillary adsorption) to a marked degree [5] The result of this surface concentration is the peculiar superficial viscosity or rigidity, first observed by Plateau [6] Vernorel and Dantony[7] were the first to point out that a liquid, exhibiting surface viscosity to a marked degree, possesses a high wetting power in consequence Thus a 1 per cent solution of saponin is capable of wetting a glass plate coated with paraffin wax, although a 5 per cent solution of soap fails to do so The wetting power of solutions of saponin, gelatin, etc, seems to depend upon this capacity to form liquid planes, the high superficial viscosity of which prevents rupture and running together to form drops

The explanation of "wetting" is, however, still obscure, and our views of the subject are in a state of transition, largely as the result of the work of Langmuir and Harkins

Both Langmuir and Harkins have developed an essentially similar theory of surface tension phenomena, thus Langmuir states "A theory of surface tension is now proposed in which a structure of the surface layer of atoms is regarded as the principal factor in determining the surface tension (or rather surface energy) of liquids According to this theory the group molecules of organic liquids arrange themselves in the surface layer in such a way that their active portions are drawn inwards, leaving the least active portion of the molecule to form the surface layer "[8]

Harkins states "That surface tension phenomena in general are dependent upon the orientation and packing of molecules in surface layers "[9]

Langmuir[10] regards the spreading of an oil on the surface of water or of a liquid on the surface of a solid as a chemical process, rather than a physical one He attributes the special surface activity of both solids and liquids to the "residual valency" or the "unsaturated chemical affinity" of the atoms The presence or absence of such will determine whether or not a liquid will spread upon a surface "The spreading of an oil upon water is thus due to the presence of an 'active group' in the molecule, that is some group which has a marked

affinity (secondary valence) for water " Thus oleic acid spreads on the surface of water owing to the presence of the carboxyl groups, whilst oils without active groups, like pure saturated hydrocarbons, as Hardy[11] has shown, do not spread

Further, Langmuir states " Consider first the adsorption of a liquid by a plane solid surface If the molecules of the liquid contain active groups, the molecules will become orientated and will pack into the surface layer in much the same manner as in the case of oil films spread on the surface of water "[12]

Whilst these views of Langmuir and Harkins throw considerable light on the process of wetting, they do not affect the relation between the three tensions concerned referred to above

A simple method of determining the wetting power of a liquid, *e g* , a tree spray, would be very advantageous, but from the foregoing it is obvious that such a determination is attended with considerable difficulty As a result, various means of comparing the wetting power of liquids have been suggested from time to time, some of the commoner of which merit passing consideration

The most commonly accepted of these is the determination of the surface tension Thus, Brunnich and Smith[13] state that " the wetting power of any liquid, or its property to form a uniform film upon a greasy surface, depends primarily upon its surface tension," and they attempt to compare wetting power by measuring surface tension by means of a stalagmometer Vermorel and Dantony[14] state that surface tension as measured by the capillary rise method affords a satisfactory criterion of wetting power Later, however,[15] they state that " the surface tension of a liquid is not sufficient to determine its wetting power The wetting power manifests itself very differently, according to the nature or state of the substance to be wetted With regard to plants, the wetting power of some solutions appear to depend less upon the surface tension than upon the surface viscosity, as defined by Plateau " Lefroy[16] also recommends a determination of the surface tension as a means of comparing the wetting power of spray washes

From the expression given above, it follows that a liquid to wet well must possess both a low surface tension to air (T_1) and a low interfacial tension (T_{12}) to the solid to be wetted To compare wetting power by a determination of the surface tension of the wetting liquid only is misleading for two reasons (1) The nature of the surface to be wetted is completely disregarded , (2) owing to surface concentration effects, the surface tension (static) of soap solutions does not vary with concentrations so diverse as 0 1 and 10 per cent (expressed as fatty acids), yet the wetting power of such concentrations differs greatly

The interfacial tension (T_{12}) is itself indeterminate, but in actual practice a workable compromise may be made by the suitable choice of a liquid to represent the surface to be wetted Thus, for testing cattle dips, tree-sprays, horticultural washes, etc , in which the surface to be wetted (hide, leaves, bark, etc) are of a greasy or waxy nature, a thick neutral mineral oil may be taken (compare Cooper and Nuttall, *loc cit*) or if the wetting power of a lubricating oil is under considera-

tion, mercury would be selected to represent the bearing surface (Compare Bhatnagar and Garner[17])

The determination of the interfacial tension of two immiscible liquids is readily carried out by means of a stalagmometer[18] For comparative purposes, the interfacial tension may be taken as indirectly proportional to the "drop number," but where absolute values are required, the method of calculation used by Lewis[19] must be employed

The figures for the interfacial tension given in the following table have been obtained by this method, those for the surface tension by Searle's torsion balance method[20]

TABLE I

Surface Tension and Interfacial Tension of solutions of Castor Soap towards Liquid Vaseline

Concentration Per cent of Fatty Acid	Surface Tension of Soap Solution T_1	Surface Tension of Vaseline T_2	Interfacial Tension T_{12}	$\Gamma_{..}-(T_1+T_{12})$
	dynes/cm	dynes/cm	dynes/cm	dynes/cm
2 0	33 45	31 11	8 23	−10 57
1 0	33 45	31 11	10 93	−13 27
0 5	33 45	31 11	11 98	−14 32
0 1	33 45	31 11	27 39	−29 73
0 01	33 45	31 11	76 46	−45 35
0 001	56 61	31 11	108 43	−77 32
0 0001	70 76	31 11	129 28	−98 17

It will be noted that whilst with concentration from 0 1 to 2 per cent of fatty acid there is no marked variation in the surface tension of the soap solution, with the interfacial tension there is a progressive decrease in value as the concentration increases It is evident also that the interfacial tension is the predominant factor in determining the wetting power of a soap solution, and much valuable assistance in the control of many technical operations of great diversity can be obtained by "drop number" determinations Unfortunately, the "drop number" method has its limitations It is only reliable in the case of soap solutions With solutions of gelatin, saponin, etc, unless these are extremely dilute, surface concentration effects at the interface of the oil and aqueous solution become so marked that quasi-solid surfaces are produced and the "drop number" completely vitiated

Another suggested means of measuring wetting power is by a comparison of the emulsifying power of the wetting liquid towards oil Donnan (*loc cit*) and Hillyer[21] have shown independently that a soap solution acts as an emulsifier by virtue of its low interfacial tension to the oil to be emulsified Since wetting power is also mainly dependent upon a low interfacial tension, it follows that a liquid, capable of emulsifying oil readily, will also possess a high wetting power

Similarly, a fine emulsion of oil in a soap solution will obviously have a high wetting power The presence of the emulsified oil does not in any way increase the wetting power, its presence in a finely-emulsified state is merely a proof that the liquid has a high wetting power

TABLE II

Surface Tension and Interfacial Tension of solutions of Castor Soap towards Liquid Vaseline in the presence of Emulsified Solar Oil

Solution contained 0 1 per cent of fatty acid plus—	Surface Tension of Solution T_1	Surface Tension of Oil T_2	Interfacial Tension T_{12}	$T_2-(T_1+T_{12})$
	dynes/cm	dynes/cm	dynes/cm	dynes/cm
2 0 per cent of Solar oil	49 08	31 11	42 46	−60 13
1 0 ,	50 00	31 11	44 47	−63 36
0 5 ,	48 19	31 11	37 06	−54 14
0 25 ,,	49 66	31 11	37 39	−55 94
0 1 , ,	48 81	31 11	37 98	−55 71
0 01 ,,	39 56	31 11	37 39	−45 84
0 001 ,, ,	33 45	31 11	27 39	−29 73

Table II gives the wetting power of solutions of castor soap containing varying percentages of emulsified oil If these figures be compared with those given in Table I, it will be seen that the emulsified solution has a distinctly lower wetting power

Foaming power has also been suggested as a criterion of wetting power Foam formation has been investigated by various workers of whom mention may be made of Plateau (*loc cit*), Quincke,[22] Rayleigh,[23] Donnan (*loc cit*) and Shorter[24] The essential conditions for the formation of foam are that the liquid shall be a solution and shall possess either the property of surface concentration, *e g*, saponin solution, which shows exceptional frothing power, or a low surface tension, *e g*, soap solution Since, however, wetting power is mainly dependent on a low interfacial tension, the ability of a liquid to give a permanent foam does not necessarily indicate a high wetting power

Of the industrial applications of wetting power, one of the most important is that of mineral separation, by which low-grade tailings, hitherto wasted, can now be profitably extracted It is computed that the method now deals with about 60,000,000 tons of ore annually [25] The process is mainly (though by no means entirely) concerned with sulphide ores, and is based upon the differences shown by various substances in the degree to which they are wetted by water or other liquid Thus water wets quartz (gangue) more readily than a mineral sulphide This difference in "wettability" is accentuated by the addition of a small appropriate quantity of a suitable oil, which, by being selectively adsorbed on to the surface of the sulphide, renders it more difficult to wet Similarly, by the addition of an acid or alkali, the surface of the quartz is made more easily wettable In the earlier

methods, the unwetted particles of sulphide were allowed to rise to the surface and skimmed off (skin flotation), in modern practice, air is forced through the mixture, when the sulphide particles are adsorbed at the surface of the air bubble and carried upwards (froth flotation) The technique of the process is beyond the scope of the present article, but Broadbridge[26] describes the process in detail, Sulman (*loc cit*) gives a detailed account of the theory upon which the method is based, as also does Edser[27]

Langmuir[28] classifies surface flotation phenomena roughly into three classes —the formation and properties of the froth, the oiling of the solid particles, and the adhesion of the oiled particles to the bubbles of the froth, and discusses these from the standpoint of his own work on oil-films, and his theory of surface tension and adsorption "The formation of froth depends on the presence of substances which can form a stable monomolecular film over the surface of each bubble" Langmuir has not sufficient data to draw any conclusions between constitution and powers of flotation of various oils, but Dean[29] claims that "flotation oils possessing a polar group are adsorbed at the air-water interface," and that "from the chemical formula of the oil it is possible to determine its flotation qualities" Langmuir (*loc cit*) states that "the adhesion of the particles to the bubbles depends primarily on the ease with which oily water wets the oily solid, and this in turn is measured by the angle of contact between these oiled liquid and solid surfaces"

Froth flotation methods are now being applied to a variety of minerals, other than sulphides, viz, cassiterite, oxides and carbonates of copper, tin oxide, lead and silver oxides

A recent adaptation has been the separation of bitumen from rock, described by Fyleman[30] Bitumen frequently occurs in association with more or less finely divided mineral matter, the content of bitumen usually ranging between 10 and 20 per cent The problem of separating the bitumen from such deposits has hitherto proved impossible for economic reasons, extraction by organic solvents or removal by distillation being too costly Washing with hot or cold water has also been attempted, but since the interfacial tension between the mineral matter and the bitumen is usually lower than that between the mineral matter and water, no separation of the bitumen from the rock resulted By employing a liquid, however, the interfacial tension of which towards mineral matter is less than that between the bitumen and the mineral matter, separation of the bitumen from the rock is readily effected

Very dilute solutions (1 part per thousand) of an alkali soap, of an alkali salt, of a weak organic acid, or of saponin serve the purpose

As most bitumens contain small amounts of compounds of a weakly acid character, a very dilute solution of sodium carbonate frequently suffices (Fyleman, English Patent, 1635, 1920) The method is also applicable for the recovery of mineral oil from the saturated sand occurring round oil wells

Another recent application of differential wetting is a separation of coal from mineral matter, but it is doubtful whether in this country

at any rate such a process could be worked profitably In Germany, a froth flotation method has been patented for the separation of graphite from its ores

A second important industrial application of wetting power is that relating to the use of contact poisons, whether as veterinary preparations, *e g*, cattle dip, or as horticultural medicaments, *e g*, tree sprays The writer has, in collaboration with Cooper, emphasised this point elsewhere [31] In tropical and sub-tropical countries, stock-breeding is only possible if constant steps are taken to keep under various insect parasites, many of which are disease carriers Of such parasites, ticks are responsible for an enormous annual loss to cattle breeders The usual method of protection is by systematic dipping in a dilute solution of sodium arsenite, but in S Africa, especially with the " Bont " tick, it has been found that the concentration necessary to kill the tick frequently leads to the destruction of the cattle through bad scalding

By incorporating an oil emulsion with the arsenite and thus insuring a high wetting power, it proved possible materially to reduce the arsenic content of the dip, and yet insure the destruction of the tick without injury to the cattle Wetting power is of equal importance with all forms of tree sprays and insecticides generally The leaf of a tree, the chitinous integument of an insect and the protective woolly secretion of the aphis or American blight are all exceptionally difficult to wet, and unless wetting occurs, a contact poison cannot do its work The well-known tree spray consisting of an emulsion of paraffin and soap solution owes its efficiency to the high wetting power conferred by the soap

The importance of wetting power in the process of scouring has already been referred to by McBain in his article on the Colloidal Chemistry of Soaps [32]

The interfacial tension, as determined by the " drop number," towards a standard oil is a convenient and satisfactory method for determining the cleansing power of soaps The temperature at which the determination is made should correspond as far as possible to that at which the soap is to be used [33]

In the degumming of silk by means of a froth bath, a low surface tension is the main requisite The increased frothing power resulting from the presence of solid colloidal matter should not, however, be overlooked Thus, for example, by diluting sodium oleate solution until no permanent lather is produced and then adding a pinch of clay, a persistent lather can be obtained [34]

Since emulsification of oils by soap and similar solutions is mainly dependent on a low interfacial tension between the oil and the emulsifying liquid, the " drop number " of the oil to be emulsified against solutions of various soaps, etc, will afford a good indication of the best type of soap for emulsifying the oil in question The " drop number " also affords means of measuring the efficiency of Twitchell's re-agent towards the oil to be saponified Twitchell's re-agent acts mainly through its power of emulsification, as a result of which the oil becomes emulsified and thus offers a greatly enlarged

surface to the hydrolysing action of the acid present [35] Other emulsifying agents such as colloidal clay can be substituted for the Twitchell re-agent and increased hydrolysis similarly obtained [36]

Anti-dimming preparations also represent another industrial application of wetting power Such compositions were largely used during the war for the eye pieces of gas masks whilst to-day they are beginning to find application for use on the wind screens of motor cars The object of such is to reduce the interfacial tension between water and celluloid or glass, so that moisture or rain is no longer deposited in the form of drops which obliterate vision, but wets the material with an even film so that vision is not seriously impaired The American Army Chemical Service carried out considerable experiments on anti-dimming compositions [37]

An instance in which the opposite effect is desired, namely, that water shall be deposited in the form of drops, is afforded by the process of shower-proofing fabrics A high interfacial tension between water and the surface of the fabric is obviously necessary and this is obtained by coating with a surface film of wax or aluminium stearate, etc With such a dressing the rain fails to wet and runs off in drops whilst the porosity of the fabric remains practically unimpaired

The addition of saponin to an aqueous liquid to enable it to spread more easily over a semi-greasy surface, *e g*, ink on writing paper, is well known

Wetting power is naturally not confined to aqueous liquids, but whenever a liquid, whether aqueous or otherwise is applied to a solid surface, surface and interfacial tensions come into play, and the spreading of the liquid over the surface or otherwise thereby determined Thus, for example, wetting power plays an important part in the paint and varnish industry Paints and varnishes during application frequently refuse to spread or sometimes, especially with spirit varnishes, as the solvent evaporates, the varnish runs into drops This effect is known technically as "cissing" Frequently it arises through the surface to which the paint or varnish is applied being damp or greasy, and in these cases is easily overcome by sand papering the surface In other cases, however, the defect is to be referred to the paint or varnish itself From what has been already said, it is obvious that the surface tension of the paint or varnish is not mainly responsible for the trouble, but rather the interfacial tension between the liquid and solid surface which is of far greater significance in the paint industry Thus H A Gardiner and P C Holdt[38] have determined the surface tensions of liquids concerned and have obtained the following averages in dynes per cm , triglyceride drying oils, 38 5 , turpentine, 31 5 , mineral spirits, 30 They have also determined the "drop number" of various paint liquids against water and claim that such measurements may apply closely to practical conditions, since all solid surfaces are believed to hold an adsorbed film of moisture Some of their results are —Raw linseed oil, 55 drops , linseed fatty acids, 300 drops , turpentine, 115 drops , mineral spirits, 79 drops Addition of linseed fatty acids to raw linseed oil greatly decreases the interfacial tension and, therefore, the wetting power

Finally, " wetting " plays some part in the process of lubrication Thus, Ubbelohde pointed out that only a liquid which " wets " a solid can constitute a true lubricant Wells and Southcombe[39] have followed up this idea and have determined the interfacial tension of various oils towards water Their results show that the interfacial tension against water of the vegetable and animal oils is much lower than is the case of a mineral oil, and they find that " the lowering of the interfacial tension against water in the case of the fatty oils was due to their slight content of free acidity " Lewis has measured the interfacial tension between oil and a liquid metal mercury, and concludes " with some confidence that the addition of the organic acids will lower the tension at any metal-oil interface " This result has been confirmed by Bhatnagar and Garner[40] who state that " it thus appears that the increased efficiency of a mineral oil to which an organic acid is added runs parallel with a lowering of tension at a metal-oil interface "

That the addition of a fatty acid to a mineral oil materially improves its lubricating properties has been shown by Archbutt [41] Determinations were made on a Thurston machine run at a slow speed to obtain contact friction and at a constant load The following results may be quoted —

	Mineral Oil	Rape Oil	Friction Coefficient
	Per cent	Per cent	
1	100 0	Nil	0 0066
2	99 5	0 5	0 0049
3	99 0	1 0	0 0045
4	98 0	2 0	0 0042
5	100 0	Nil	0 0066

Similarly, Hyde working with a Deeley machine at the National Physical Laboratory found that the addition of 0 1 per cent of rape oil fatty acids added to a mineral oil lowered the coefficient of static friction from 0 132 to 0 092, a reduction of 30 per cent [42] Archbutt[43] concludes " that when a lubricating oil wets the surface of a bearing, a layer one molecule thick becomes absorbed by the metal and forms part of the solid surface This absorbed film entirely changes the physical character of the surface and lowers the static friction. The best lubricants are those most active in this respect, and the mineral oils deficient in oiliness can be improved by dissolving in them substances whose molecules are more active This, in my opinion, is the true explanation of the remarkable effect caused by adding small quantities of fatty acids to mineral oils " As an outcome of Wells and Southcombe's work, " straight " mineral oils containing small additions of suitable fatty acids are now displacing the mixtures of mineral and vegetable oils hitherto used as lubricants A considerable saving of valuable vegetable oils has thus resulted

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COLLOIDS IN RELATION TO THE MANUFACTURE OF INKS

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It is a remarkable fact that the first application of Colloid phenomena to industrial processes should have been in connection with the manufacture of ink In the preparation of Chinese or Indian ink—an account of which, dating back to about B C 2600, is extant[1]—lamp black is obtained by burning an oil such as Chinese wood oil with a limited

supply of air, and is then pounded with a solution of glue or gum and made into sticks which are dried

In order to obtain a pigment that will remain in suspension for a long period, the smoke is sometimes fractionated by being passed through a series of chambers, and the deposit which is formed in the last chamber is used for the finest qualities of ink

If equal weights of different qualities of Indian ink are ground up in equal volumes of water, pronounced differences will be noticed in the rates at which the carbon particles subside, and it is possible in this way to grade samples rapidly into different classes

In a paper recently published by Lucas[2] interesting information on the flocculating properties of modern Egyptian carbon inks is given

Kuhn[3] adapted the physiological method of counting blood corpuscles to the examination of finely powdered pigments A weighed quantity of the material is mechanically shaken with a measured quantity of a medium such as glycerol or linseed oil, a measured quantity of the mixture withdrawn by means of a micro-pipette, diluted with the medium, and the number of particles counted in a Zeiss-Thoma chamber under a magnification of 550 diameters By this means it was found that lamp black with a specific gravity of 1 57 contained 960 milliards of particles per gram, and from this it was calculated that an average particle had a diameter of 10 8μ, and contained 26 milliard molecules of carbon

The addition of a carbon pigment, such as lampblack, has often been suggested as a means of rendering ordinary writing ink proof against the action of chemical agents, but in most cases added lampblack subsides more or less rapidly, owing to its not being in a sufficiently fine state of division to form a homogeneous mixture with the solution of the soluble pigment The same objection applies to the addition of ordinary graphite, as first claimed by Halfpenny (Eng Pat 262, 1873), but this difficulty has been largely overcome by the invention of Acheson's deflocculated graphite, which is now used as the carbon pigment in many so-called " safety " inks

The ordinary writing inks of the present day consist of a more or less soluble tannate of iron, prepared by mixing together an infusion of galls, myrobalans or other source of tannin, with copperas or other salt of iron

When this type of ink was first made, it was exposed to the air until it gradually changed into a black colloidal tannate which remained in suspension in the liquid, and gave a dark colour to it In the more modern inks, typified by the so-called " blue-black " inks, the iron tannate is kept in a more soluble condition by the addition of a suitable proportion of hydrochloric or other strong acid When this ink is applied to the paper the oxidation takes place gradually, and successive oxidised tannates are produced, ending finally in the formation of an insoluble tannate Pending this darkening process the writing at first gets its colour from an added dye, such as indigo or aniline " soluble blue "

The stages of this change from a soluble iron tannate, through a colloidal tannate, into ultimately an insoluble tannate, have been studied by Mitchell[4] who has shown that to this change must be attributed the different behaviour of inks in old and recent writing to the action of chemical agents

When ferrous tannate solution is exposed to the air the precipitate which is first produced contains about 5 5 per cent of iron and is of a colloidal character, it corresponds with an iron tannate described by Wittstein [5] The final insoluble precipitate contains approximately 8 per cent of iron, corresponding with a tannate described by Pelouze [6] Between these two extremes come precipitates with intermediate proportions of iron, which are possibly mixtures of these two compounds

The addition of a suitable amount of acid is necessary to keep the ink in a stable condition of arrested oxidation, and if the acid is reduced below a definite amount the ink assumes a colloidal character and eventually throws down heavy deposits It has been shown by Mitchell[7] that these objectionable changes may be caused by alkali in the glass neutralising part of the acid in the ink, and that this is probably the cause of many previously mysterious deposits in bottled ink

Difficulties caused by changes of a colloidal character are also observed in the case of other inks For example, copper logwood ink is often very unstable, and chrome logwood is liable to coagulate suddenly Stein[8] claimed that the addition of mercuric chloride would liquefy the coagulated ink, but Viedt[9] found this remedy to be ineffective, and obtained better results by adding sodium carbonate and preventing the ink from coming into contact with the air

In the case of marking inks, examples of colloidal preparations are to be found in some of the silver inks, although in others the silver salt is probably present in true solution and is then reduced to a black compound, possibly, an oxide, by the action of heat Some of the aniline marking inks are still made in two solutions, one containing the aniline salt, and the other the oxidising agent, and these are mixed immediately before use to form a colloidal solution, which when heated yields an insoluble deposit of aniline black

There are also numerous preparations in which the two essential constituents are present in one solution, and remain fluid until the ink is applied to the fibres, when the volatile substances present evaporate, and the insoluble black pigment is formed

Some of these inks contain an excess of free aniline over the oxidising agent, whilst others contain both free aniline and acetic acid, both of which evaporate on exposure This is the principle adopted in Grawitz's marking inks (Fr Pat 276,397, 1898), but unless the proportions of the constituents are properly balanced the change will take place in the bottle, and the ink, after remaining fluid for months will suddenly gelatinise into a viscous mass, which is useless for marking purposes

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THE MANUFACTURE OF ARTIFICIAL SILK IN RELATION TO COLLOID CHEMISTRY

By EDWARD WHEELER, A.C.G.I. A.I.C.

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INTRODUCTION

The manufacture of artificial silk furnishes a very good example of an industry where the technique is considerably in advance of the theory of the process. Many of the minor operations involved in making the solutions, coagulating the thread, etc., have been found to be of fundamental importance in that they effect changes in the colloid state of the cellulose which ensure the production of a commercially successful fibre, and have been introduced largely as the result of what might be termed empirical research. Gradually, however, the methods of colloid chemistry applied to the study of the technical problems of the industry are revealing the scientific principles underlying these operations, and leading to further important improvements and developments in manufacture. Unfortunately, much of the technical research work carried out by manufacturers of artificial silk and other cellulose products remains unpublished for obvious reasons, but it is to be regretted that work dealing with the more general aspects of cellulose and regenerated cellulose could not be published, as much work in this direction has had to be carried out in the research laboratories of many firms. The patent literature affords some indication of the directions in which technical research is being prosecuted, but little information is there afforded as to the scientific or technical value of the discoveries thus protected.

Processes employed for the Manufacture of Artificial Silk

The four processes for the manufacture of artificial silk in use to-day are as follows —

(*a*) Cuprammonium process (also known as Glanzstoff or Pauly process)
(*b*) Nitrocellulose process (also known as Chardonnet process)
(*c*) Viscose process
(*d*) Cellulose acetate process

The bearing of colloid chemistry on each of these processes will be briefly discussed in relation to the raw materials used, the preparation

of the solutions, the coagulation of the thread in spinning, and the after-treatment necessary in each case The properties of artificial silk as a colloid will then be considered in relation to the requirements of the textile industry

Cellulose as the Basis of all Artificial Silk

The basis of all commercial artificial silk at present being manufactured is cellulose, but only cotton and wood-pulp cellulose (sulphite pulp) have been utilised successfully as raw materials The former is used in the cuprammonium, nitro-cellulose and cellulose acetate processes , the latter in the viscose process

Cotton represents the highest grade of cellulose obtainable, and it is therefore not surprising to find that the best artificial silk has been made from cotton Nevertheless the product produced from wood-pulp cellulose by the modern viscose process has approached very closely to that produced by the other processes It should, however, be mentioned that owing to its cheapness the viscose process has largely supplanted the other processes both in this country and on the Continent for some years, and hence the methods of manufacture have been much further improved and developed by research and experimental work, so that to this reason may be ascribed the high grade product thereby produced Recent work has shown that a viscose silk made from cotton is superior in many respects to the wood-pulp derived product

(a) Cuprammonium Process

The cuprammonium process depends on the solution or dispersion of cellulose in a copper ammonia solution and the coagulation of cellulose hydrate in the form of a thread by expressing the solution into acid or alkaline baths of suitable composition

Preliminary preparation of the cotton —The dispersion of raw cotton in cuprammonium solution is a very slow process, and the maximum content of cellulose which can be obtained in solution is not more than 4 per cent[1] Such a content of cellulose is too low for practical purposes, and moreover the viscosity of such a solution is too great for spinning purposes It is, therefore, necessary in the first place to free the raw cotton from impurities and depolymerise the cellulose uniformly by kiering it under certain standard conditions (such as strength of alkali, time of kiering, kiering pressure, etc), and if this treatment is not sufficient the kiered cotton is mercerised or gently bleached whereby the depolymerisation of the cellulose complex is carried further, and solutions of higher cellulose content and having a viscosity more suitable for spinning are obtained Each manufacturer has worked out his own conditions[2] and little information has hitherto been available on this point During the war the whole question of kiering cotton was very thoroughly investigated by the Ministry of Munitions in connection with the production of nitro-cellulose The effect of such factors as concentration of caustic soda, the temperature and time of boiling, on the viscosity of the cotton in cuprammonium solution were determined in the case of a large number of samples

of cotton As a general result of this semi-large scale investigation[3] it appeared that although considerable variations might exist in individual samples of cotton, under standard conditions of kiering, cotton can be freed from impurities and depolymerised uniformly to a certain stage *

Preparation of the cuprammonium solution —The cotton thus treated is dissolved in copper-ammonia solution usually prepared by blowing air or oxygen through ammonia in contact with copper in a suitable form The patent literature dealing with the preparation of copper-ammonia solutions is very extensive, and a large number of different methods of making the solution are protected [4] According to Berl[5] the colloidal portion of the cuprammonium hydroxide unites with the cellulose to form an adsorption compound soluble in ammonia Various patents have been taken out to ensure the preparation of the maximum amount of colloidal copper hydroxide,[6] as thereby the solubility of the cellulose is increased The usual amounts of copper, ammonia and cellulose in the commercial spinning solutions range between 2 5–3 0 per cent copper, 7–8 per cent ammonia, and 7–8 per cent cellulose

A systematic investigation of the copper-ammonia, cellulose equilibrium was made by Connerade[7] who proved that the fixation of copper as colloidal copper ammonium hydroxide by the cellulose obeys the adsorption law, and that the solubility of the cellulose brought about by this fixation extends right up to the limit of equilibrium between the solid and liquid phases The strongly hydrated colloidal complex also fixes ammonia in proportion to its concentration tending towards the stabilisation of the solution

The solutions of cellulose in copper-ammonia thus obtained are, however, not stable , firstly, they undergo oxidation by direct absorption of oxygen from the air whereby the cellulose is oxidised,[8] and the viscosity of the solution rapidly decreases , secondly, the cellulose sol tends to revert to the gel state In order to render the solution more stable, three methods have been tried[9,4], which may be classified as follows —

(*a*) The addition of an organic hydroxy compound, *e g* , sugars, starch, etc

(*b*) The addition of caustic soda in small amounts

(*c*) Preparation and preservation of the solutions at low temperatures (0° to 6° C)

The effect of adding substances in class (*a*) is, from the chemical point of view, to prevent oxidation of the cellulose at the expense of the added compound, though at the same time their function may be partly that of a protective colloid in increasing the stability of the cellulose sol , while the effect of caustic soda (*b*) is possibly to increase the stability of the colloidal copper-ammonia complex and so to increase

* It has long been known among manufacturers of artificial silk that the viscosity of the cellulose solution bears a very definite relation to the strength of the fibre made from it and as far as possible it has been the endeavour of the manufacturer to keep the viscosity within certain arbitrary limits found to be consistent with satisfactory spinning and high strength of the product

the stability of dispersion of the cellulose itself,* the effect of low temperature (*c*) on cellulose in contact with alkaline or acid solutions of certain strengths is always to increase its gelatinisation, whereby it passes more easily into solution or dispersion, while the rate of change of sol-gel is considerably retarded

The solution of cuprammonium obtained is not spun directly, but is usually kept for some days According to Foltzer[10] three to four days ripening is sufficient, but on the other hand in some cuprammonium factories periods of two to three weeks are not unknown During this period of ripening a slow change in the solution takes place as is shown by the decrease in viscosity of the solution, pointing to a depolymerisation or breakdown of the cellulose complex by the solvent or the dispersoid In practice it is found best to keep the solution for a definite period at a uniform low temperature, namely, about 4° C

The solution is carefully filtered† and evacuated during the ripening period prior to spinning

Spinning.—The spinning process consists in forcing the solution by means of air pressure or of a pump through a number of fine glass jets, or one steel jet bored with several holes (each hole being about 0 1 to 0 2 mm diameter), into a fixing bath, one filament being formed from each hole A certain definite number of these filaments are combined together to make up a single thread according to the size required ‡ The size of the thread to be spun determines the amount of solution of known cellulose content projected through the jet or jets by means of the pump and the rate at which the composite thread is drawn off by the collecting spool per minute

Coagulating or spinning baths.—The coagulating or spinning of the thread from the cuprammonium solution can be effected by (*a*) acid, or (*b*) alkaline baths, the former type of bath has been largely superseded in recent years by the latter

Before considering these types of baths it is necessary to specify shortly what properties are required in the finished thread, so that a proper understanding of the action of the different constituents of the two types of bath may be arrived at A commercially satisfactory artificial silk should possess high strength—dry and wet, elasticity,

* The effects produced by these additions have been claimed in one form or another in various patents over a number of years Each specific addition is claimed to add some special property to the 'silk' spun as regards strength and lustre but undoubtedly the general effect is for the cellulose to be present in a more finely divided or dispersed state or continuous homogeneous phase in a stable solution whereby a more homogeneous thread is spun While with a thread spun from a solution verging on the gel state, or discontinuous phase, a loss of cohesion will result on spinning the thread so that the particles as precipitated do not tend to hold together so continuously and hence an inferior thread is produced The inferiority of such a thread is shown by its low mechanical strength—wet and dry tenacity—and poor elasticity

† The filtration of cellulose solutions for spinning artificial silk has to be effected most efficiently In a recent patent the use of high speed centrifugals (15,000 r p m) has been claimed as a means of eliminating the very small undissolved particles (*Brit Pat*, 171,384)

‡ Counts of artificial silk thread are termed deniers The denier is defined as the weight in milligrams of nine metres of "silk" In most artificial silk the size of individual filaments is seven to eight deniers (*see* p 68)

softness, good covering power and lustre Softness and lustre will not be so directly influenced by variation in the spinning bath, as the other properties and the aim of all manufacturers, other things being equal, has been to produce a thread having the highest possible strength and maximum covering power The covering power will obviously be dependent on the cross-sectional area of the threads As a result of continued research the types of spinning baths which yield an artificial silk having maximum covering power with the highest strength and elasticity have gradually been evolved

Bearing these considerations in mind it will be obvious that the cross-section of the thread must be largely influenced by the method of coagulation as conditioned by the composition temperature and time of action of the spinning bath (This latter factor will depend on the length of travel of the formed thread through the bath and the rate at which it travels through it to the winding spool) Moreover, as in many other chemical reactions, it might be expected that a difference would be observed between the action of an acid or alkaline bath alone and baths containing in addition salts or organic bodies In fact it is by the addition of these other constituents to the baths that the rate of coagulation and the rate of penetration of the bath through the initially-formed film can be favourably influenced, so that a satisfactory thread is produced [11]

(*a*) *Acid spinning baths* — The usual type of acid spinning bath formerly adopted consisted of 50 to 60 per cent sulphuric acid with or without other constituents acting at ordinary temperatures, weaker acid was found to give a thread lacking in solidity, softness and strength

(*b*) *Alkaline spinning baths* —Alkaline spinning baths have been found to coagulate the thread more slowly, whereby a more even thread is produced possessing greater covering power A typical bath contains 16 to 30 per cent caustic soda and 7 per cent sugar

The patent literature dealing with alkaline spinning baths is very voluminous, but the majority of baths patented are only modifications as regards strength, organic additions, etc , whereby it is claimed that a thread of special properties is obtained Generally, it would seem that the effect of an alkaline bath is to precipitate the cellulose in a more gel-like form yet the thread is coagulated sufficiently to prevent separate filaments sticking together By such alkaline baths a very highly hydrated open thread is obtained which is soft and has good covering power An acid fixed thread is less gel-like and therefore possesses less markedly the required properties, though it is possible to modify the rapid coagulating action of the acid by the addition of salts, etc , to the bath

The use of alkaline precipitating baths has made possible the production of threads of much finer counts (down to 0 49 deniers per filament) than could be produced by any other process, as the threads, being in a more plastic condition, can be drawn out to a very considerable extent without breaking Thus coarse threads are spun from steel jets having holes of 0 75 mm diameter into a 0 25 per cent caustic soda bath, and then are subjected to a drawing-out

process while being gradually passed by an ingenious arrangement into a bath of 5 per cent caustic soda (Thiele process[12, 13])

In this connection reference might be made to an investigation on the formation of threads in spinning cuprammonium solution from glass jets, which has been carried out by Ost[14] He has studied the formation of the thread at the jet and its drawing out in gel state using different types of capillary jets He found that a thread spun from a 0 2 mm jet could be drawn out up to five to six times its original length provided that the strength of the spinning bath was so arranged that the thread was in a plastic gel condition

After-treatment of the spun thread —The thread produced from an acid or alkaline bath is in the former case washed free from acid, and in the latter case fixed first in 5 to 10 per cent sulphuric acid and then washed free from acid The fixed thread is now an irreversible colloid, but in a highly hydrated condition It is necessary in order to produce the bright and lustrous artificial silk to dehydrate this thread in such a way as to prevent contraction on drying This is usually effected by drying the thread at 40° to 60° C by a current of warm air (care being taken to keep the humidity below 50 per cent), sometimes out of a soap solution, on suitable stretching frames whereby no contraction of the thread takes place, and a lustrous thread is obtained It might be here remarked that within limits it is possible to stretch the skeins or threads slightly in the process whereby an enhanced strength may be obtained at the expense of the elasticity of the thread

The bright thread thus produced can be further treated by bleaching, dyeing, etc, but any subsequent treatment does not affect appreciably the permanent nature of the cellulose hydrate produced after the first drying

Recovery of the copper —The commercial success of the cuprammonium process, apart from the production of satisfactory artificial silk, depends almost entirely on the successful recovery of the ammonia and the copper used in the process From the point of view of colloid chemistry the most interesting methods are those concerned with the recovery of the small amount of cuprous oxide in the large volume of water used for washing the spun thread This has been successfully carried out by the addition of various insoluble colloids, such as starch or finely-divided cellulose, whereby the colloidal oxide is adsorbed and so can be easily recovered[15]

(*b*) *Nitro-cellulose Process*

The nitro-cellulose process of manufacturing artificial silk consists in dissolving nitrocellulose in suitable organic solvents and forcing the solution thus obtained through glass jets, having suitable orifices, into a spinning bath (wet spinning) or into warm air (dry spinning) whereby the solvent is evaporated The thread thus produced consists of nitrocellulose, which has to be denitrated to render the silk safe for commercial use

Preparation of Nitro-cellulose Solution —The manufacture of nitrocellulose from the colloidal point of view has already been referred to in previous reports[16]

Cotton is always used as the raw material for the manufacture of nitro-cellulose for artificial silk manufacture, as it has hitherto been found that the viscosity of a solution of nitro-cellulose made from wood-pulp is too low for successful spinning [17] On the other hand, the exigencies of the war period caused considerable research to be carried out in the production of a wood-pulp suitable for smokeless powder manufacture Satisfactory results were obtained especially in Germany, and it seems possible that wood-pulp cellulose might be used for the production of nitro-cellulose for artificial silk [17a] According to Schwalbe and Becker[17a] the freeing of the wood cellulose for nitro-cellulose manufacture from the impurities usually associated with it can be successfully accomplished by a lime boil without depolymerising the cellulose to any extent The work carried out in this country by the Ministry of Munitions (see page 51) showed that the conditions of kiering cotton profoundly affected the viscosity of the nitro-cellulose in ether-alcohol mixtures, the usual solvent used in artificial silk manufacture

The first stage of the mixed process consists in nitrating suitably purified cotton at 40° C , with an acid having the following approximate composition H_2SO_4—44 per cent , HNO_3—38 per cent , H_2O—18 per cent , the degree of nitration being determined by examination of the nitrated cotton under the microscope [18] The nitrated cotton is washed and purified by methods well known in the explosives industry, and then partially dried and dissolved while still moist in ether-alcohol (60 40) By dissolving the moist nitro-cellulose in ether-alcohol it has been found that a more fluid and soluble solution can be obtained than by using anhydrous nitro-cellulose , moreover, it has the advantage of rendering the process less dangerous

Gibson and McCall have carefully studied the viscosity of nitro-cellulose solutions in ether-alcohol, and have found that there is a definite relation between the viscosity and the percentage of alcohol in the ether-alcohol mixture, the viscosity usually passing through a minimum at a definite ether-alcohol ratio Moreover this relationship holds for different concentrations of nitro-cellulose, but varies for different percentages of nitrogen [19] Masson and McCall studying mixtures of acetone, water and nitro-cellulose found that a similar relationship held [20]

The nitro-cellulose solutions are very stable, the viscosity decreasing very little in 21 days, but after six months a diminution takes place [21] According to Masson and McCall (*loc cit*), the viscosity of a nitro-cellulose solution rises to a maximum within a few minutes of making the solution and then falls very gradually both with aqueous and anhydrous acetone or with ether-alcohol as solvents The process of solution consists, firstly, in the absorption of the solvent by the nitro-cellulose accompanied by the dispersion of the resulting gel , secondly, in a change in the already dispersed gel, which may be ascribed to a change in aggregation of the particles With ether-alcohol mixtures, the absorption of solvent with resulting swelling of the particles and decrease in volume of the surrounding fluid continues for some time even after primary dispersion has occurred

Various additions to nitro-cellulose solutions have been proposed, but they have proved of little value or interest from the point of view of artificial silk production

Spinning—There are two well-known processes of coagulating the thread produced from nitro-cellulose solutions, viz, (*a*) wet spinning process, coagulating by liquids, (*b*) dry spinning process, coagulating by evaporation of solvent The latter process is the one now in most general use, but the former is of interest from the colloid point of view, as it enables a comparison to be made in respect of the formation and cross section of threads produced by two entirely different methods [22]

(*a*) *Wet Spinning Process*—In this process a relatively dilute solution of nitro-cellulose (10 per cent) is spun into water or dilute acid, etc [23] The thread thereby, as in the cuprammonium and viscose processes, is to a certain extent permanently fixed on issuing from the jet or jets, and the coagulum being fairly dilute the shrinking on drying is large and results in a considerable contraction, giving a thread possessing a star-shaped cross section and so having good covering power

By spinning into dilute alcohol, the hardening of the surface layer is prevented and the gel-thread shrinks at the same rate and produces a cylindrical thread [24]

(*b*) *Dry Spinning Process*—In this process a strong solution (18–25 per cent) is expressed from suitable glass jets into a current of warm air, by this means the circumference of the thread is not fixed before the interior has solidified The cross section of such threads is of a kidney shaped contour Such a thread has less covering power, but otherwise has proved to be satisfactory At the same time it is necessary to allow the evaporation of the solvent not to be too rapid either by controlling the temperature of hot air (45° C to 70° C) or the speed of spinning, and at the same time to control the moisture so as to prevent the deposition or condensation of moisture in the thread whereby a dull thread is produced By slow evaporation of the solvent at the start, the moisture first deposited in the thread is removed by the time coagulation is completed, and the thread is soft and lustrous [25]

Solvent Recovery—The recovery of the solvents evaporated in the dry spinning process is of great importance, as upon its success depends the economic working of the nitro-cellulose process The recovery of ether-alcohol was fully investigated during the war in connection with the manufacture of propellant explosives, and a process was worked out depending on the solubility of these vapours in cresol [27] One of the most interesting methods recently proposed is that of Levy[27] who claims the use of activated charcoal as an absorbent for the ether-alcohol vapour

Denitration—The thread spun from the solution of nitrocellulose in ether-alcohol must be converted into cellulose before it can be handled as a commercial product Although the exact mechanism of the nitration process is not yet completely elucidated[16], yet there would seem to be no doubt that the effect of the nitration process

must, to a certain extent, depolymerise the cellulose complex, though the depolymerisation will be partially masked by the entry of $-NO_2$ groups The removal, therefore, of the attached $-NO_2$ groups will give a cellulose of less complexity than the original cellulose Cellulose (regenerated) from its esters always exhibits less complexity than the cellulose from which it has originated as can be shown by the fact that it has a lower viscosity in cuprammonium solution Various methods have been suggested for denitration, but the most successful process is that in which sodium hydrosulphide is used (10 per cent) at 35° C , whereby the alkaline hydrolysis is slow, easily controllable, and involves least attack of the cellulose [28]

The nitrogen content of the denitrated thread cannot be reduced below 0 05 per cent , without destruction of the thread Besides traces of nitric esters, traces of esters of sulphuric acid are left in the thread which have been the cause of rotting of the thread developing on storage [29] The question of the occurrence and elimination of these esters of sulphuric acid has been fully investigated in connection with the manufacture of nitro-cellulose explosives Their elimination has been very largely effected by the steaming stabilisation process, pulping and poaching of the nitro-cellulose, methods which cannot be adopted in the case of artificial silk manufacture Evidence tends to show it is necessary to hydrolyse these esters so that most of the combined sulphuric acid, which leads to instability in explosives and acid tendering in the silk, is eliminated, leaving a residue which does not give rise to these objectionable defects [30] The cause of the development of these acid patches in artificial silk, after the alkaline denitrating process it has undergone, must be ascribed to insufficient washing after nitration, or washing with water containing calcium salts whereby unstable and insoluble salts are adsorbed within the thread and gradually decompose giving rise to free sulphuric acid The difficulty with which sulphuric acid is entirely eliminated from cellulose textiles is well known, and must be ascribed to the formation of a sulphuric acid-cellulose adsorption compound

After treatment —The denitrated silk is soured, bleached, stretched and dried in the usual manner

(*c*) *Viscose Process*

At the present time the greater portion of the world's annual production (approximately 10,000 tons) of artificial silk is produced by the viscose process Several descriptions of the main lines of the process have been published[12, 31], but as these descriptions are lacking in those details which are not only of interest from the point of view of colloid chemistry, but are also important for the success working of the process, it is necessary to describe the different operations specially involved at some length

Raw Materials —Viscose can be made from any form of cellulose, but for artificial silk manufacture only bleached sulphite wood-pulp and cotton have been successfully employed (*cp* , page 51)

The bleached sulphite wood-pulp cellulose used must be almost free from lignin[31a] and colouring matter, and must be only bleached

itly that its content of oxycellulose is low (as indicated er reduction figure)—and by the Cross and Bevan test— of α-cellulose should be at least 80 per cent In cotton test α-cellulose is usually of the order of 95 per cent he cellulose in wood pulp is similar in constitution to is still an open question, but in the writer's experience eem to be no doubt that the cellulose complex is more highly polymerised in cotton than in wood pulp This is shown by the fact that under standard conditions viscoses obtained with cotton are always of a higher viscosity than those obtained with wood pulp moreover, stronger artificial silk is produced therefrom

Preparation of the Viscose Solution —The preparation of the viscose solution involves three distinct operations, viz —

(*a*) Mercerisation ,
(*b*) Xanthation ,
(*c*) Mixing and ripening

(*a*) *Mercerisation* —The cellulose whether in the form of wood-pulp sheets or combed cotton fibre is immersed in aqueous caustic soda (17 5 per cent) at normal temperatures for three to six hours, whereby the threads or fibres are completely swollen The excess of liquor is pressed out and the cellulose-soda complex disintegrated to a bread-crumb like mass having a surface about three-and-half times that of the original pulp This mass is usually kept loosely packed in bins at 20° C—25° C for 24 to 72 hours, whereby the cellulose-soda complex undergoes a change, oxygen being absorbed from the air The cellulose is depolymerised and the viscosity of the resulting viscose influenced, so that by the regulation of the time and/or the temperature of this so called mercerisation any viscosity of viscose made therefrom required within limits can be produced In practice most wood-pulps require about 50 hours at 23° C to give a solution of suitable viscosity for spinning, and to enable a satisfactory thread to be produced It has been found that the oxygen absorption can be accelerated by the presence of catalysts in the form of certain metallic oxides or hydroxides [32]

(*b*) *Xanthation* —The mercerised alkali cellulose is next treated in a revolving drum with carbon disulphide whereby the bread-crumb-like material is gradually (churned) converted into a yellowish brown rubber-like mass In practice it is very important that the xanthation process should be stopped just before the whole mass tends to lose its individuality and become one solid gelatinised mass From experiments carried out by the writer it seems that the viscosity of the resultant viscose to a certain extent depends on the stage at which xanthation is stopped, lower viscosity values being obtained invariably when the cellulose xanthate is "over-churned" During xanthation there is a distinct rise in temperature (about 10° C to 12° C), and at the same time oxygen is being absorbed The carbon disulphide is distributed between the cellulose-soda complex and the free soda As the cellulose xanthate is formed, or as more and more CS_2 molecules enter the cellulose aggregate, the temperature rises

which causes some of the cellulose xanthate to decompose, CS_2 being split off and combining with the free soda to form thio carbonate, sulphide, etc , hence the importance of stopping the "churning" process as near as possible to the stage when the maximum of xanthation has taken place with the minimum formation of by-products

A recent patent[33] has claimed the use of a so-called colloid mill whereby the cellulose, soda and carbon-disulphide are brought into such intimate contact (colloidal) that a purer viscose is formed free from thio salts and does not require ripening (*see* below), and is supposed to give silk having improved strength and dyeing properties

(*c*) *Mixing and Ripening*—The cellulose xanthate is dispersed in weak caustic soda solution whereby the rubber-like solid gelatinises and gradually passes into solution in a few hours The solution thus obtained is carefully filtered two or three times during the ripening period and then is ready for spinning

Reference has already been made in the Second Report to the ripening of viscose, but the phenomenon is not discussed in detail As pointed out above, cellulose xanthate itself undergoes decomposition almost as soon as it is formed, especially with a rise in temperature When the cellulose xanthate is dispersed in the caustic soda the change does not cease but continues all the time, the rate of decomposition (hydrolysis) being increased by a rise and decreased by a fall in temperature To a certain extent this change is hydrolytic the xanthic residues being gradually split off from the cellulose aggregate, so that the ratio of cellulose to sulphur gradually increases till no combined sulphur is left Before however, this stage is reached, the whole mass would gel owing to the diminishing solubility of the lower xanthates in the soda solution In practice, therefore, it is essential to spin the solution at such a stage that the resultant silk has the desired properties from the manufacturing point of view Generally, for a new viscose the cellulose sulphur = 1 75 $C_6H_{10}O_5$ 2 S, while at the spinning stage, *i e* , after ripening cellulose sulphur = 3–4 $C_6H_{10}O_5$ 2S

Many patents have been taken out for additions to the viscose solution which are either supposed to confer some special property on the resulting artificial silk or to dispense with or shorten the ripening period , in actual practice, however, these additions are of little value It is of interest however to summarise some of the various additions that have been suggested and the effects produced by them as they throw some light on the changes that viscose undergoes in ripening

Addition	Effect Produced
Extra caustic soda	No specific effect
Sodium sulphate	No specific effect, the ultimate solidification of the viscose takes place sooner owing to the salting out effect of the salt
Ammonium sulphate	Neutralises the caustic soda, and diminishes the solubility owing to formation of ammonium cellulose xanthate

Addition	Effect produced
Glucose or other hydroxyl compounds	Greatly accelerates the rate of decomposition of the cellulose xanthate
Urea and cyanamide	Preserves the viscose, retarding the rate of decomposition
Sodium sulphite	Retards the rate of decomposition of the cellulose xanthate

Sodium cellulose xanthate is an unstable colloidal body reverting to cellulose hydrate The means by which it is decomposed in practice into cellulose hydrate—artificial silk—will be discussed in the next section, but it seems that the changes that it undergoes in ripening are very complex, and cannot wholly be ascribed to the simple hydrolysis of the cellulose xanthate by caustic soda [24]

Spinning —The earliest patents dealing with the spinning baths for the preparation of artificial silk from viscose emphasised the necessity for spinning the viscose at a definite stage in its composition, whereby it was claimed the best silk was obtained The object for this lies in the fact that the higher the degree of xanthation, that is the more there are of CS_2 molecules combined to the cellulose, the more soluble is the cellulose xanthate, consequently its precipitation or coagulation in the form of a permanent thread will largely depend upon the strength of the coagulating agent This coagulation can be effected in three ways—

(1) By salt baths ,
(2) By changing the sodium cellulose xanthate into a less soluble compound ,
(3) By decomposition of the xanthate into cellulose hydrate

The first two methods are only of scientific interest, as all the present manufacturing processes are based on the third method It might, however, be mentioned that a salt bath throws down the cellulose xanthate when new in a suspensoid state which only slowly coagulates on standing Ammonium and zinc sulphates will give the insoluble ammonium and zinc cellulose xanthate, and although the formation of the former compound served as the basis of the original process of manufacture of artificial silk from viscose in this country, its use was soon abandoned

Decomposition of the xanthate is effected for spinning purposes by means of acids or acid salts whereby cellulose hydrate is directly regenerated With a new viscose even a strong acid acts but slowly owing to the very highly dispersed condition of the cellulose xanthate, so that it is almost impossible to spin a new viscose of a high degree of xanthation Gradually, however, the viscose in ripening reaches a stage where it can be fixed or coagulated sufficiently to be drawn away from the jets Generally viscose can be spun after a ripening period of 30 to 140 hours, at 17° to 20° C , with acid ranging from 20 to 8 per cent H_2SO_4 This, of course, only refers to the actual coagulating action of the acid, as acid alone is never used, but always a mixture of sulphuric acid and salts with or without organic additions The function

of the salt or salts is to restrain the violence of the acid coagulation o the thread sufficiently to allow it to be spun successfully [35]

It might be mentioned here that three other factors necessarily have to be considered in relation to the coagulating effect of the acid, etc, namely, the temperature of the spinning bath, the length of immersion of the spun thread from the orifice of the jet to the point at which it leaves the bath and the speed at which it is drawn through the bath The correlation of these factors with the hydrogen ion concentration and the coagulability of the different salts used would probably explain the specific differences claimed for the very empirical baths set forth in the patent literature It is obvious that the longer the spinning bath acts on the thread, either by variations in the length of immersion or in the speed of spinning, the greater will be the fixing effect The usual spinning speed in the Topham* spinning process is from 30 to 45 metres per minute, while the immersion can range from 2 5 to 25 cm varying with the denier required and with the individual preferences of different firms (a recent patent has claimed a speed of 76 metres per minute[36]) Another interesting direction in which viscose spinning baths have been developed is in the addition of certain organic compounds to the bath, which exert a very beneficial effect The most usual organic compound used is glucose but any sugar or hydroxyl compound seems suitable The function of the organic compound in the viscose spinning bath has been explained in many different ways, and the case of the use of glucose may be specially discussed [37] The amount used is generally between 8 to 10 per cent, in presence of 8 to 10 per cent sulphuric acid and 12 to 14 per cent sodium sulphate Primarily the glucose may be said to act mechanically by interfering with the action of the acid in so far as its fixing effect is concerned, and also possibly osmotically by hindering the penetration of the acid through the initial film of cellulose hydrate formed at the jet It has also been claimed that glucose keeps the thread moist and so prevents crystallisation of the sodium sulphate within the pores of the thread before it is reeled into skeins It may also increase the solubility of the gases of decomposition in the spinning bath so that a thread free from any pitting effect is obtained Undoubtedly, the effect of its addition to the spinning bath has been of very great importance in the spinning of viscose, as it has enabled a more flexible and less harsh thread to be produced

There is also a connection between the composition of the spinning bath, temperature and the rate of decomposition of the thread, *i e*, cellulose xanthate—cellulose hydrate It is generally found best to carry out the spinning at 35 to 45° C as at this temperature spinning can be effected at a reasonable commercial speed It is, however, very noticeable that the colour of the spun thread after leaving the spinning bath changes sharply from yellow to white when the temperature of the spinning bath exceeds 55° C

* In the Topham process the thread is spun and drawn off by a glass pulley and transferred through a funnel into a centrifugal revolving at 2500–3000 r p m, by which means the thread is coiled and given the necessary twist

at a spinning speed of 35 metres per minute It is well-known that it is quite possible to spin a thread which is quite soluble in water directly after spinning although it becomes insoluble (irreversible) on standing [38] Presumably, therefore, there is a definite temperature at which the thread is completely decomposed or rendered insoluble for any given set of spinning conditions (immersion, rate of spinning, composition and temperature of bath, etc)

Besides the addition of organic compounds to the bath reference might be made to patents which have been taken out for using baths such as have been described but also containing small quantities of zinc sulphate [39] It is claimed that the addition of zinc sulphate in quantities not exceeding 1 per cent has a considerable effect in increasing the dry strength of the thread The reason for this is not obvious, but the explanation may be found in a modification of the film of cellulose initially formed at the jet whereby certain different osmotic effects, from those obtained when zinc sulphate is not present, are brought about The property of films in *statu nascendi* are unknown, differences in tenuity, permeability, etc , may easily be brought about by small differences in composition of the spinning bath which may profoundly modify the properties of the finished thread

Reference has already been made to the production of fine filaments of artificial silk (from 0 5 to 2 deniers per filament, *vide* p 54) from cuprammonium solutions by drawing out a coarse thread Hitherto the production of such fine threads by the viscose process has not been possible commercially but in a recent series of patents [40] Bronnert has described methods whereby such fine filaments can be produced merely by the use of spinning baths of special composition Hitherto the chief difficulty in spinning fine counts with viscose has been the lack of elasticity of the initial thread formed at the jet especially if the slightest tension is put upon it in attempting to draw it out To overcome this Bronnert proposes the use of baths of sulphuric acid of such a concentration that they are inversely proportional to the square root of the denier In the first of the series of patents (Brit Pat 163817) he claims baths containing 360 grams Na_2SO_4 per litre with 120 to 140 grams H_2SO_4 for 7 5 deniers per filament, 170–190 grms H_2SO_4, for 4 deniers, 250–280 grms H_2SO_4 for 2 deniers, the rate of spinning (40 to 50 metres per minute), size of jet orifice (0 1 mm diameter) and age of viscose (90 hrs) being the same in each case It is also claimed that a similar effect is also obtained without the salt (Brit Pat 166931), while in a third patent a mixture of ammonium sulphate and sulphuric acid is employed, the ammonium sulphate being present in an amount equivalent to half its weight of sulphuric acid and inversely proportional to the square root of the density In several other patents the same principle is claimed, in some cases the acid varying and in others certain special organic additions being claimed

According to statements made by Bronnert in the patents finer threads are obtained with stronger acids, because the initial tendency of the viscose is to form globular drops owing to its superficial tension Upon seizing and drawing out this drop the precipitating power in the

bath has a coagulating effect The skin which is thus formed on the outside surface of the thread is the less absolutely resistant the smaller the surface which is in contact with the acid, *i e*, the finer the thread is If, however, higher concentrations are used in spite of its small superficial area the corresponding film or skin apparently acquires a sufficient tenacity to be spooled without breakage, the optimum concentration of coagulant being obtained in accordance with the relationship referred to above This discovery is of great interest as indicating a definite relation between surface formation of thread and concentration of coagulating agent and illustrates how the quantitative study of colloid precipitation has led to the solution of a technical problem which has been the subject of investigation by manufacturers for several years

After-treatment of the spun thread—The thread after spinning is washed free from acid and dried under tension whereby the lustre is obtained This lustre is roughly proportional to the stretch given to the thread, and is limited by the power of the individual filaments to stand the strain Generally it is sufficient to prevent the threads contracting on drying (*Vide also* p 54)

The thread after drying is desulphurised, bleached, soured, washed and dried

(*d*) *Cellulose Acetate Process*—Attempts to utilise cellulose acetate for the manufacture of artificial silk have been frequently made during the past twenty years, but hitherto the commercial development of this process has not proved successful From the chemical point of view it resembles the nitro-cellulose process in that the thread spun consists of a cellulose ester, which is subsequently hydrolysed to cellulose hydrate If the thread of cellulose acetate could be utilised as such it would be infinitely superior in strength to any artificial silk manufactured, but unfortunately the very property that confers on the cellulose acetate its superior strength, owing to its being practically waterproof, renders it almost incapable of being dyed by the ordinary dyeing methods Hence arises the necessity for the complete or partial removal of the acetyl group

Preparation of the cellulose acetate and its solution—A very complete bibliography of the methods of making the different cellulose acetates has been compiled by Worden [41] Cotton is used as the raw material and is acetylated by means of acetic anhydride in the presence of a suitable catalyst The differentiation of the cellulose acetates depends on differences in their solubilities in acetone and chloroform, such differences in their solubility can be brought about by the so-called hydration of the acetate, either during the process of acetylation by warming the acetylating mixture at a certain stage or by subsequent hydrolysis treatment The effects produced by this hydration process would seem to indicate that to a certain extent hydroxyl groups replace acetyl groups in the cellulose complex, whereby an alteration in the solubility of the acetate is produced

After the acetate has been freed from the acetylating mixture, it is dissolved in a mixture of organic solvents, as, for example, a mixture of methyl alcohol and tetrachlorothane Numerous mixtures of solvents

have been patented and their success depends on their cheapness and the ease with which they can be recovered

As in the case of other solutions of cellulose or its esters the viscosity of the solutions thus obtained depends very largely on the previous treatment of the cellulose, the conditions of acetylation and hydration (*vide* above), and also on the composition of the solvent,[44] but provided standard conditions are adopted for the solvents, a relation is traceable between the other factors and the strength of the finished product

Spinning.—The spinning of cellulose acetate is usually effected by the wet spinning process, that is to say, the cellulose acetate is projected into baths of suitable organic liquids which precipitate the thread and retain the solvent, aqueous baths, alkaline and acid, have been employed. There is at present little information available on the setting and coagulation of the thread, though the general considerations connected with the spinning of cellulose solutions, already summarised under the other processes, apply in the case of this process.

After-treatment.—The after-treatment of the cellulose acetate thread depends on the properties required of it. Any treatment which partially or completely removes the acetyl groups depolymerises the cellulose complex (*cp* denitration, p 57) and so reduces the strength, but the dyeing properties then become similar to those of the viscose or cuprammonium threads. Thus for partial saponification the use of alcoholic soda or thiocyanate solutions has been suggested.[42] In a recent paper[43] Briggs has maintained that such methods of partial saponification can be so regulated that a thread consisting of an outer layer of 20 per cent of regenerated cellulose and an inner core of 65 to 70 per cent unaltered cellulose acetate can be obtained. This method would seem to have effected an alteration in the proportion of free OH groups within the molecule, which, according to Sproxton, may also condition the solubility properties of cellulose esters.[44]

General properties of artificial silk

The properties by which the value of artificial silk for textile purposes is judged may be summarised as follows:—

(1) Tensile strength (dry and wet) and elasticity
(2) Lustre
(3) Softness
(4) Evenness
(5) Covering power

(1) *Tensile strength or tenacity and elasticity*.—One of the most important properties of artificial silk is its strength or tenacity on which depends its weaving properties and durability. The chief aim of the artificial silk manufacturer is to produce a silk with the maximum strength and elasticity.

The relation of the strength and elasticity to the size of thread and number of filaments has been carefully studied by Dreaper and Davis[45] who, as a result of their researches on cuprammonium silk, found that the tenacity of the thread increased as the size decreased, whilst

its elasticity diminished. moreover these effects were increased with an increase in the number of filaments As a conclusion of their work they put forward the theory that, each individual filament must possess a skin and the strength of this skin and the relation of the surface of this skin to the cross-sectional area of the thread determines the strength, being greatest when the proportion of skin is greatest, and diminishing as the proportion of skin area decreases The elasticity will be least when the proportion of skin area is greatest, and will increase as the proportion of skin area diminishes According to Herzog and Jancke as a result of Rontgen ray examination, artificial silk consists of an irregular felted mass of crystalline fragments of unchanged cellulose [46*]

Whatever view is accepted as to the structure of the artificial silk thread, manufacturing experience has shown that the strength depends on the original cellulose used and the preliminary treatment to which it is submitted (*cp* p 51) the solution process, the composition of the spinning bath used, the rate at which the thread is coagulated and the degree of stretching of the spun thread As has already been mentioned, artificial silk produced from cotton has generally been found to give the best results

In the following table are given the results of tenacity and elasticity tests of some typical samples of artificial silks manufactured by the different processes

Strengths of some Artificial Silks made by different Processes

Process	Raw Material	Dry Tenacity	Wet Tenacity	Extension
		Grams per denier	Grams per denier	Per cent
Cuprammonium	Cotton	1 3†	0 5	12 5
,,	,,	2 16‡	—	14 1
Nitro cellulose	,,	1 4†	0 36	7 5
,,	,,	1 48§	0 31	15 5
Viscose	Wood-pulp	1 40†	0 55	17 0
,,	,,	1 75†	0 75	14 0
,,	,,	1 30§	0 17	13 3
Natural Silk	—	2 5	2 0	21 0

The changes effected in cellulose in the manufacture of artificial silk undoubtedly can influence the strength of the finished product favourably or otherwise, but little information is available as to the

* The recent patents taken out by Bronnert for the production of fine filaments from viscose largely depend on the formation of a strong skin which will enable the thread to withstand the pull on it during spooling after being coagulated in the bath Hitherto the ordinary spinning baths have not been able to give a film of sufficient strength to enable fine filaments to withstand the strain of spooling

† Wilson [12]

‡ Dreaper and Davis [45]

§ Tests carried out by the author, 1921

influence of any one factor directly on the strength, though certain generalisations can be made

Thus the viscosity of the various solutions used for spinning artificial silk is undoubtedly a criterion of the quality of the thread produced therefrom, it being generally admitted that a considerable fall in viscosity connotes a like fall in strength of the artificial silk produced therefrom

Again the rate of coagulation of the thread by the different baths to a large extent determines the tenacity and elasticity of the finished product

The influence of the stretching process on the strength of artificial silk shows, as might be expected, that increased stretching gives a slight increase in tenacity at the expense of the elasticity The usual practice is to carry the stretching process far enough to give the required lustre It is interesting to note that if considerable tension be put on the thread between the point of emergence from the spinning bath and the spooling or coiling box, the strength of the thread can be similarly increased at the expense of its elasticity, due undoubtedly to the fact that, at this stage, the thread is in the gel condition and is thus very susceptible to tension

So far mention has only been made of the dry strength of the thread, but one of the greatest disadvantages of artificial silk has been the great reduction in strength (approximately 60 per cent) it undergoes on wetting [46a] In the case of artificial silks having a low dry tenacity the percentage decrease in strength on wetting is even greater Generally it has been found that low wet tenacity values coincide with exceptionally low viscosity solutions

The great affinity cellulose in all forms has for moisture suggests that a cellulose-water complex may be formed of little cohesion, for when the water is replaced by formaldehyde (sthenosing*) or acetyl or nitro groups enter the molecule the cellulose or its ester is partially or completely waterproofed, and has a much higher tenacity (dry and wet)

(2) *Lustre* —The effect of the stretching process on lustre is very great If spun artificial silk is not stretched, but allowed to dry completely, so that the thread is allowed to contract as far as it will, it is found that the product is lustreless It would, therefore, appear that this is due to the breaking up of the surface of the thread so that the light is scattered, while in stretching the lustre of the thread is produced by the fact that the light is reflected from the surface In this connection it is interesting to note that if the thread is transparent in appearance it is much less lustrous, though it may have been stretched as much as another thread not so transparent, but possessing more filaments to a given size of thread and being more regular in diameter [47]

* The ' sthenosing ' process—condensation of artificial silk with formaldehyde—has been the subject of several patents (Beltzer *8th Int Congr Appl Chem* IVA, 7–21) The treatment increases the dry and wet tenacity of the thread, but alters its dyeing properties

(3) *Softness*—The softness of the finished artificial silk depends almost entirely on the flexibility of the thread and on the number of filaments to a given size, the larger the number of filaments the greater the softness The number of filaments that go to make up the composite thread is limited by the practical difficulties of spinning Usually the average is practically one filament to every eight deniers, though with the Thiele cuprammonium process (*vide* p 55) an artificial silk has been spun in which the proportion was one filament to 0 5 denier [45]

(4) *Evenness*—By the evenness of artificial silk is chiefly implied its dyeing evenness, though it may also refer to the evenness with which the threads take up salts from solution, *e g*, for the loading of the thread in incandescent mantle manufacture

One of the great difficulties found with artificial silk is its susceptibility to changes in evenness owing to slight modifications in the manufacturing process

(*a*) *Dyeing Evenness*—It is not proposed here to discuss the theory of the dyeing process in relation to cellulose which has already been dealt with from the point of view of colloid chemistry in previous reports,[48] but only to indicate briefly how variations in the dyeing of artificial silk may arise in manufacture

As a rule regenerated cellulose has a greater affinity for all classes of dyestuffs than cellulose, *e g*, cotton, while those processes of manufacture which involve the use of less highly polymerised celluloses (wood-pulp cellulose as opposed to cotton) or of more drastic chemical reagents deviate most in dyeing affinity from ordinary cellulose Thus denitrated cellulose artificial silk has a greater affinity for basic dyestuffs than either viscose or cuprammonium artificial silk This is partly due to the nitration process *per se* and also the subsequent denitration process Viscose artificial silk again has a greater affinity than cuprammonium artificial silk for such dyestuffs, but if made from cotton it more nearly resembles the cuprammonium product in dyeing properties [49]

With regard to other classes of dyestuffs differences between the processes are not quite so noticeable, but nevertheless intrinsic differences in artificial silk made by the same process and even in some cases from the same batch of solution are observable when variations are made in the spinning baths, *e g*, whether there is a difference in dyeing affinity for certain specially sensitive cotton dyes of cuprammonium silk spun in an acid or an alkaline bath Various other modifications in the process add to the total effect in causing variations in the evenness of different batches of silk, hence it has been found essential in practice to pay the greatest attention to small details in the process used to ensure that the product is made under such standard conditions that it dyes evenly

Such differences in the dyeing properties of the finished artificial silk must actually be due to variations in the adsorption capacity of the thread caused by differences in the surface of the thread and thickness of the individual filaments

Attention might be drawn to the work of Minajeff who investigated the distribution of substantive and sulphide dyestuffs on cuprammonium artificial silk, Muller and Slassarski who investigated the behaviour of cuprammonium artificial silk and crystal violet and Clement who compared the dyeing behaviour of artificial silks made by the nitro-cellulose, viscose and cuprammonium processes and ascribed the differences found to the content of oxycellulose, and in the case of the nitro-cellulose product also to the residue of nitrogen always remaining after denitration [50]

More recently Wilson and Imison have studied[51] the evenness of viscose artificial silk to the various cotton and sulphide dyestuffs at present on the market, and classified the dyestuffs according as they dyed viscose artificial silk, as even, moderately even, and uneven They ascribe the differences in evenness to dyeing to especial sensitiveness of certain dyestuffs as well as to small variations in the viscose process

So far no mention has been made of the dyeing affinity of cellulose acetate silk, though an allusion was made to the problem involved (*vide* p 64) and to processes involving the complete or partial removal of the acetyl groups (*vide* p 65), so that the thread becomes more like cuprammonium silk in its dyeing affinities Briggs[43] (*loc cit*), however, pointed out that special dyestuffs and methods of dyeing can be used for dyeing cellulose acetate and referred to the work of Clavel who has developed the technique of dyeing cellulose acetate in Switzerland Thus, for instance, cellulose acetate thread adsorbs the primary amines and simple aminoazo compounds with avidity, the adsorbed base can be diazotised *in situ* and the dyestuff developed by coupling with suitable compounds

The behaviour of cellulose acetate is, therefore, quite different from that of the other silks, and the problems involved in the development of its dyeing capacity may throw quite a new light on dyeing problems

(*b*) *Salt Absorption*—The adsorption properties of artificial silk are of considerable interest in connection with the manufacture of incandescent mantles, as it has been found that such mantles are tougher and more elastic than natural fibres Unfortunately, mantles made from artificial silk do not absorb such large quantities of rare earths from solution and do not shrink enough Nevertheless, artificial silk has been and is largely used and methods are being patented for overcoming the difficulties of adsorption [52]

(5) *Covering Power*—Reference has already been made to the differences produced in artificial silk by the various processes due to the use of one or other types of spinning bath From the weaver's point of view one of the most important properties of artificial silk is its covering power (*i e*, maximum covering capacity for minimum weight of thread) This will obviously depend on the cross sectional area of the individual filaments, and on the number of filaments which go to make up the composite thread The latter condition (*cp* Dreaper and Davis[45]) has a practical limit for the manufacturing processes adopted, though modern methods of manufacture are

tending to allow an increase of the number of filaments to a given size Provided other properties of the silk are the same, the variations in the spinning baths suggested and patented have been introduced largely with the object of increasing the covering power of the thread by altering the rate of setting or by increasing or diminishing the pull on the thread during spinning, or in other words effecting modifications in the thread while in the gel state

Thus in the case of cuprammonium artificial silk spun in a soda sugar bath, the cross section of the filament is spherical, in the case of viscose in an acid bath cylindrical, but in an acid and salt bath the sphere is corrugated, with the addition of glucose the section is rendered elongated and crinkled In the nitro-cellulose process, a thread having a kidney shaped section is obtained with a smooth outline, while in the case of the finest artificial silk made from viscose or cuprammonium the threads are oval in section [53]

All these cross sectional areas are conditioned by the coagulation of the gel whether cellulose hydrate or cellulose ester

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NOTE.—In this book will be found a complete summary of the patent literature for all processes up to 1920 For more recent patents the Journal of the Society of Chemical Industry can be consulted Foltzer's book[2] and Worden's books on 'Technology of Cellulose Esters' (1921) (Vol I, parts i, iii v), also contain very complete patent bibliographies up to 1921

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